3-dimensional microstructure characterization of porous ceramic matrix composites by X-ray tomography and FIB-slicing as database for numerical modelling

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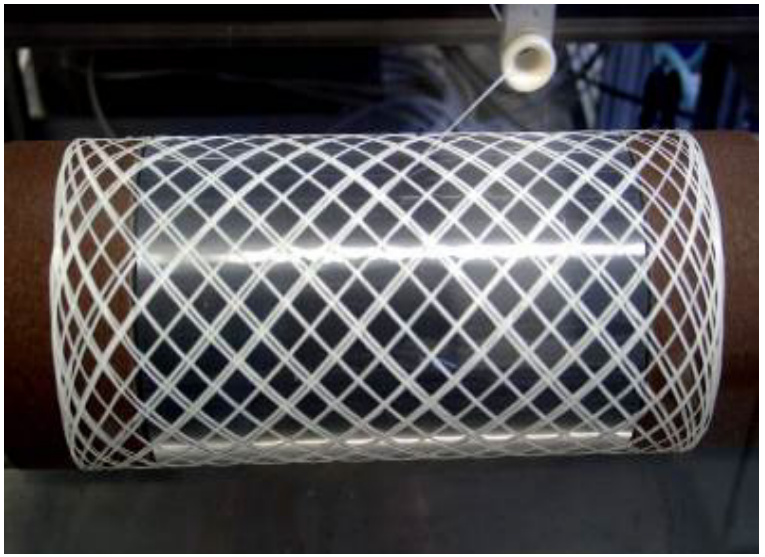
Research Goals

- Visualization of 3d meso- and microstructure of fiber reinforced porous all-oxide CMC's
- Visualization of cracks and other defects, generated during processing, testing or in service - without producing artefacts
- Generating numerical models from 3d-Images of real microstructures

CMC- Material – WHIPOX™

Wound **Hi**ghly **Po**rous **Ox**ide

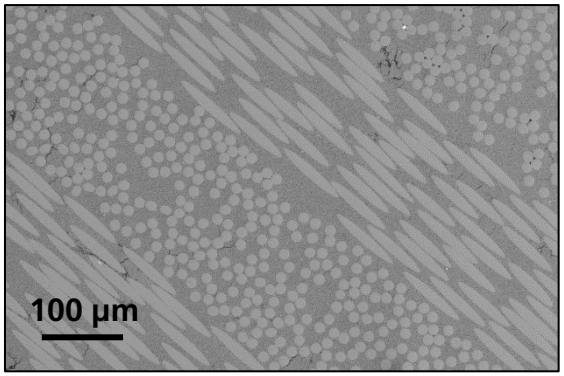


Combustion chamber for small gas turbine

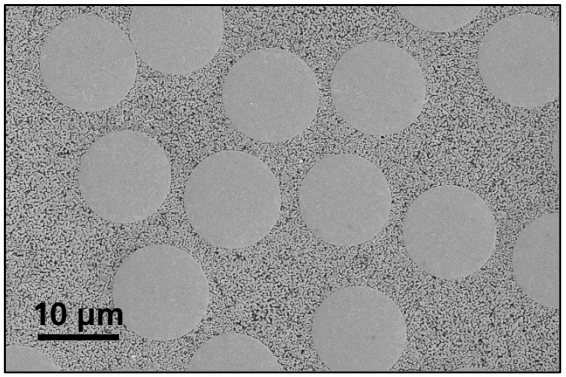
Processing by winding of alumina fiber bundles infiltrated with aqueous slurry of alumina powder and subsequent sintering

Characteristic length scales of WHIPOX™

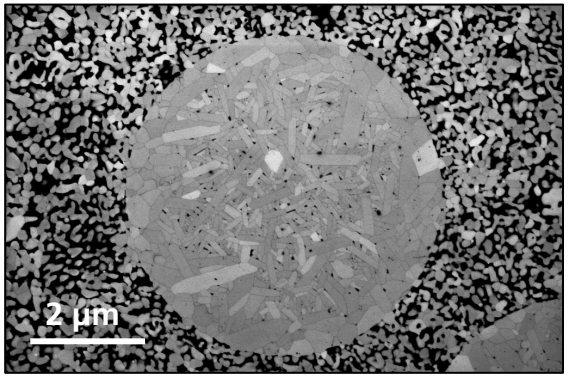
Diamond pattern of wound fiber bundles (mm – range)



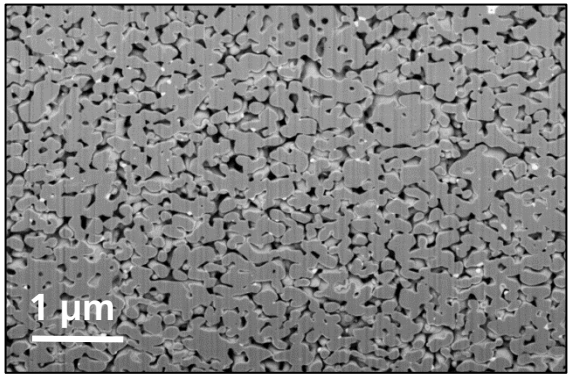
Cross section of adjacent fiber bundles with different orientation



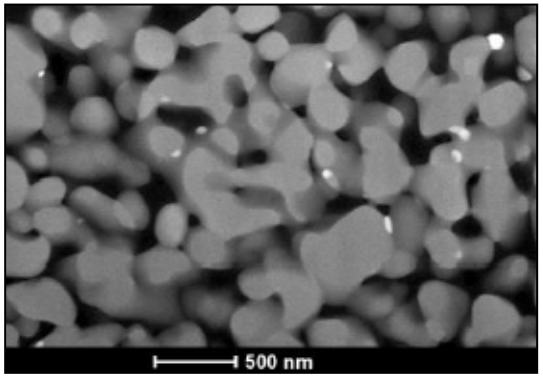
Fibers embedded in porous matrix



Single fiber embedded in porous matrix

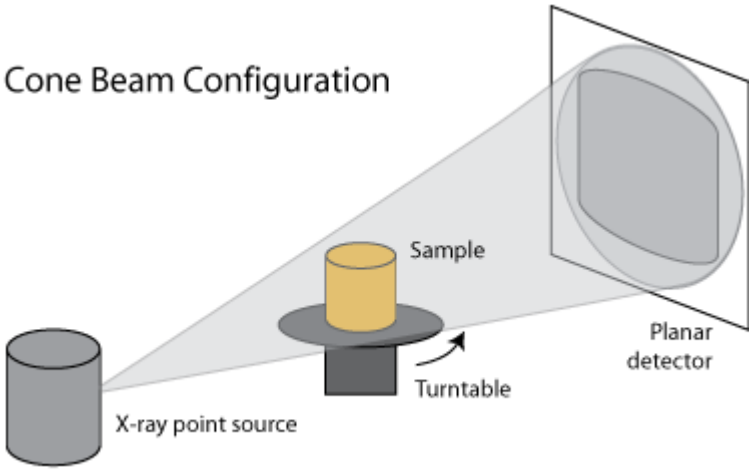


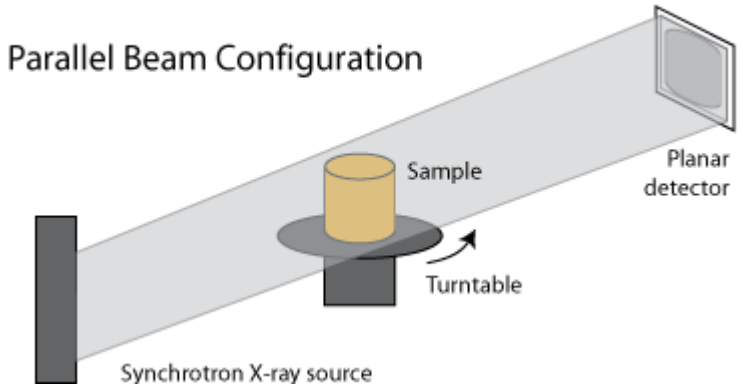
Microstructure of porous matrix



TEM-image of porous matrix (courtesy of M. Müller, GFE an der RWTH Aachen)

Non destructive generation of 3d-images by X-ray tomography





→ Computing 3d-reconstruction from set of X-ray projections of rotated sample (stepwise from 0° to 360°)

Experimental set-up at Argonne Advanced Photon source

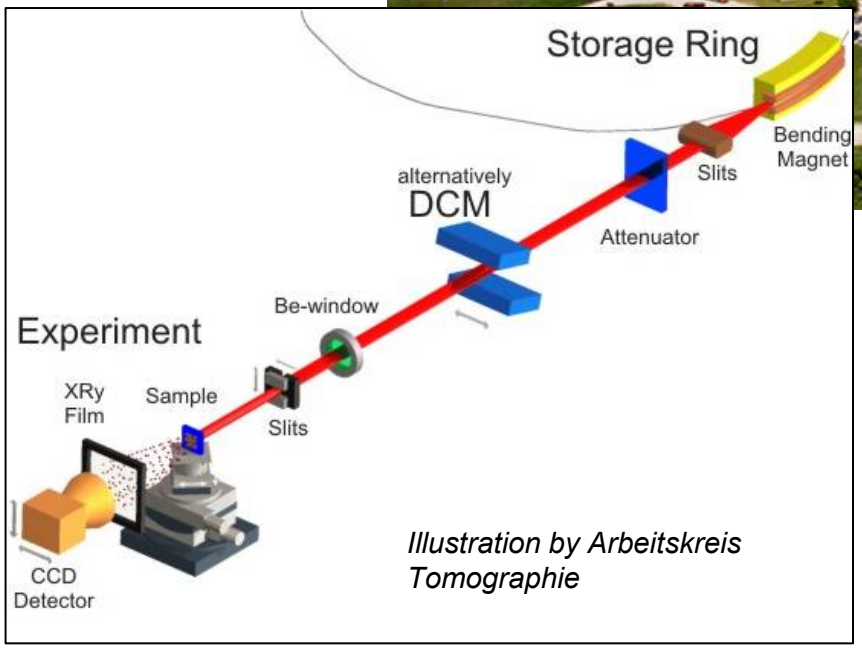
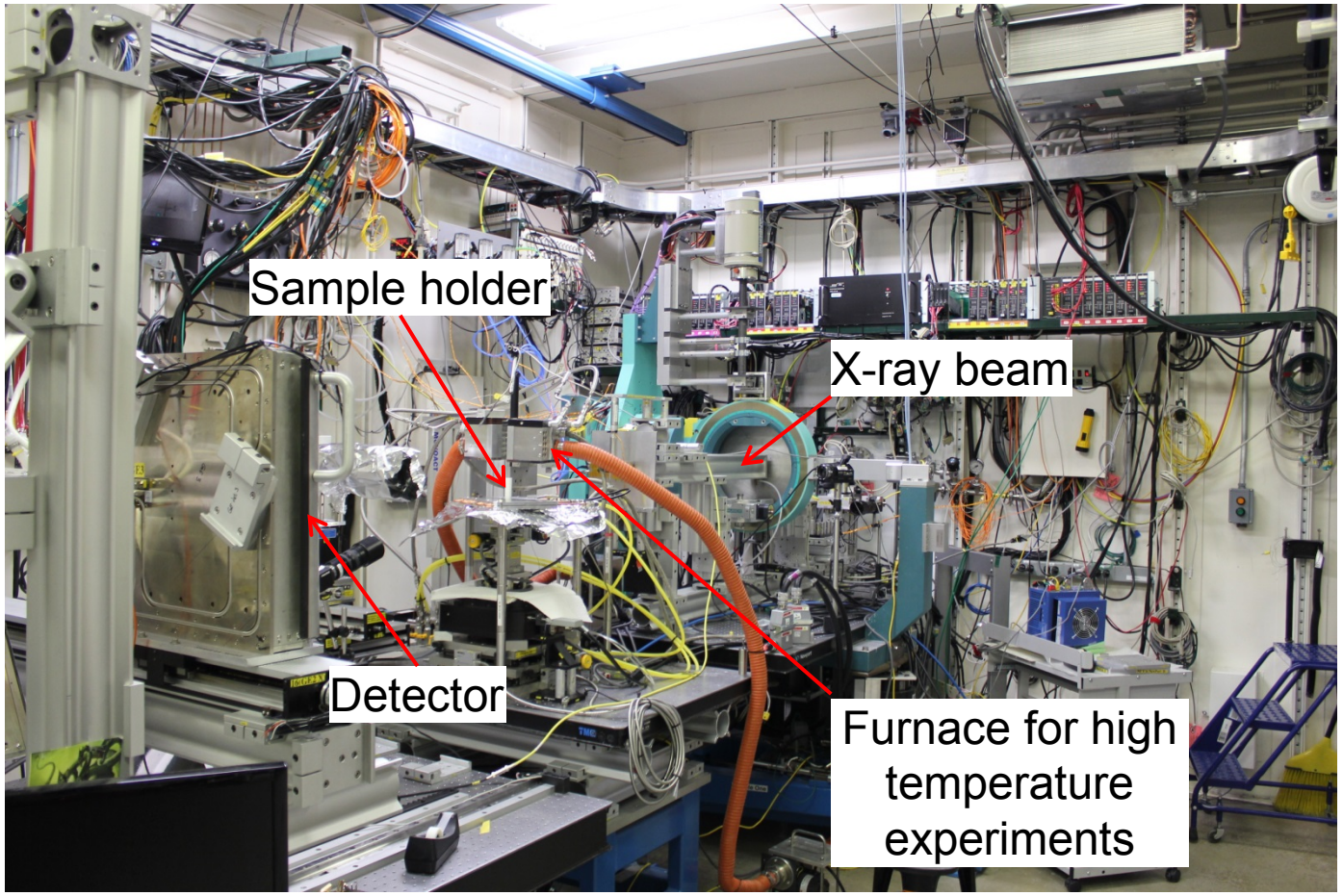


Illustration by Arbeitskreis Tomographie

- Argonne National Laboratory, Argonne, Illinois
- Synchrotron high energy X-Ray beam-line; 65keV beam energy

DLR

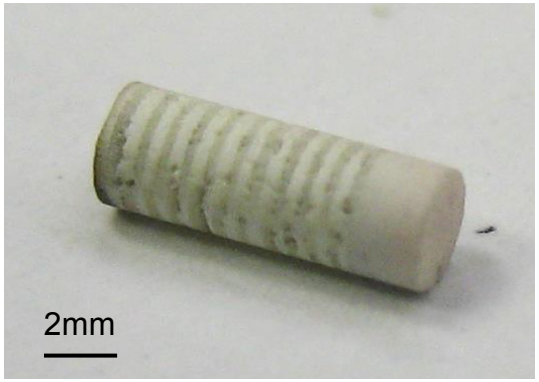
Tomography Stage for Data Acquisition



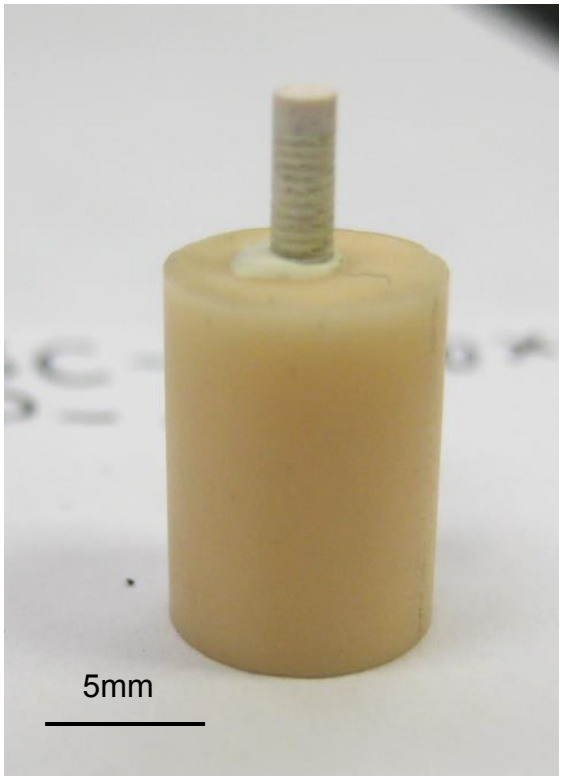
Beam width: 1.8mm

Sample preparation and mounting

Ultrasonic drilling

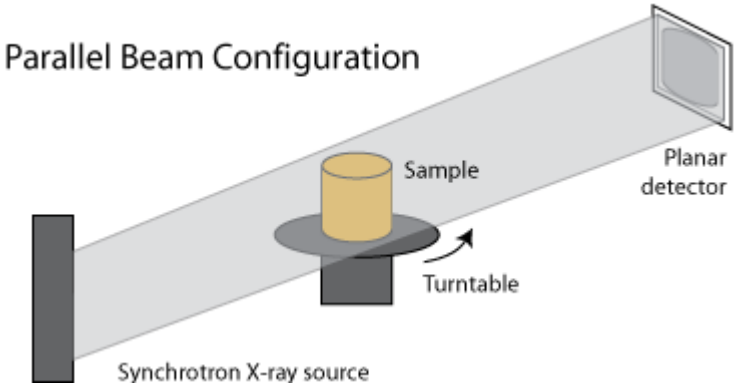


Sample with +/- 45° fiber bundle orientation

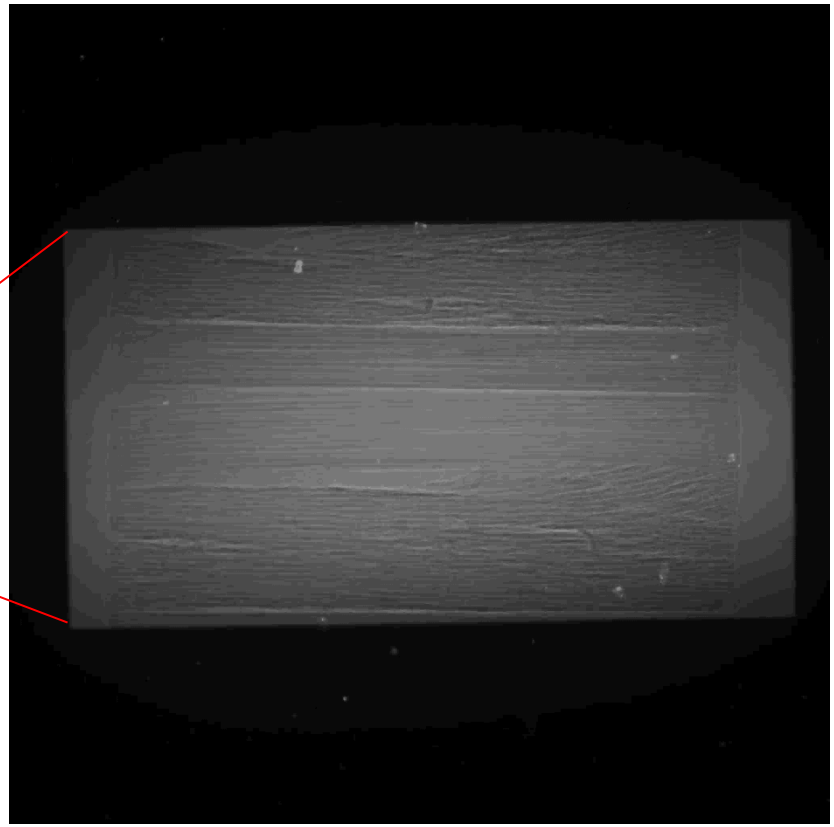


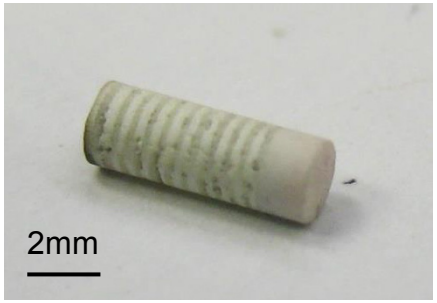
Mounted sample on high temperature and room temperature fixture

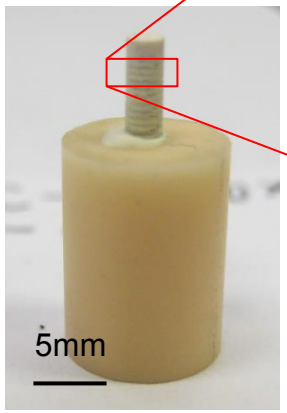
Monitoring sets of X-ray projections



Laminate with +/- 45° fiber bundles







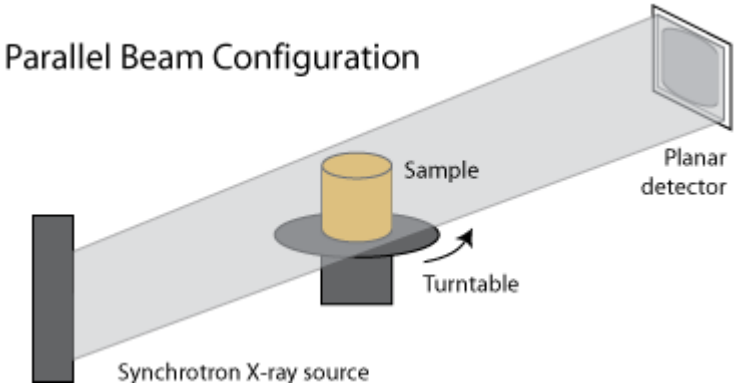
Sample

Mounted Sample

X-ray projection

DLR

Monitoring sets of X-ray projections



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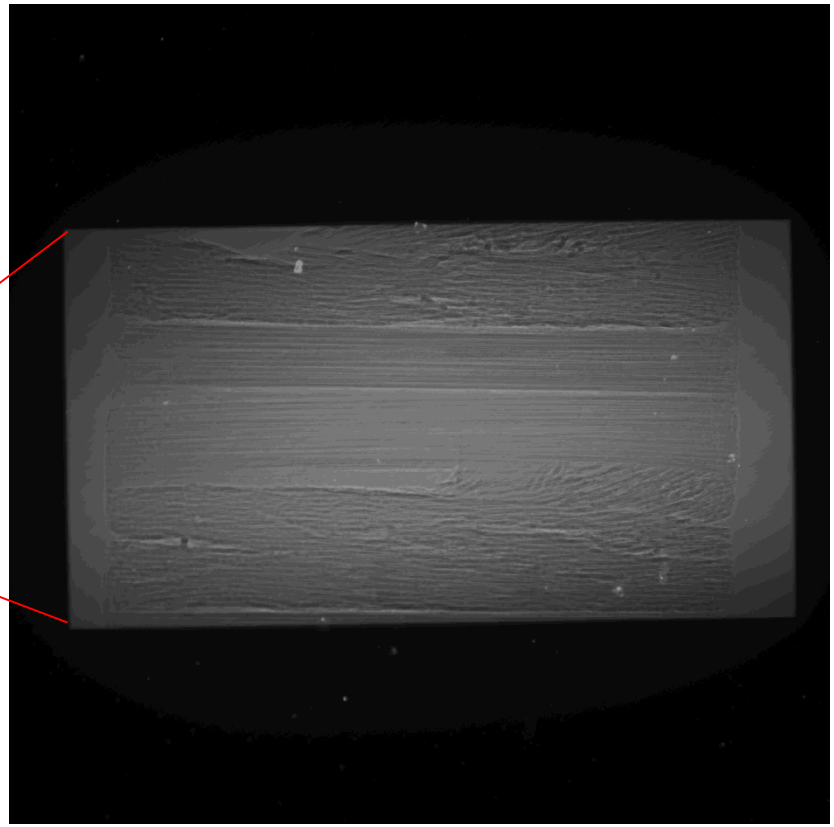

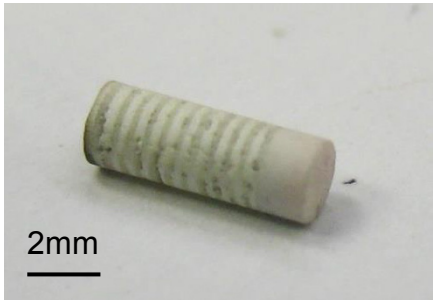

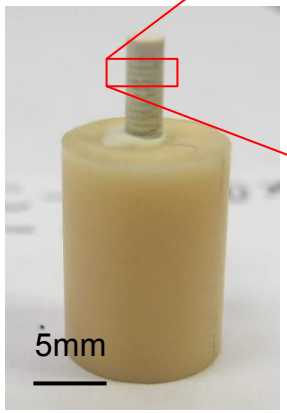

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Mounted Sample

X-ray projection

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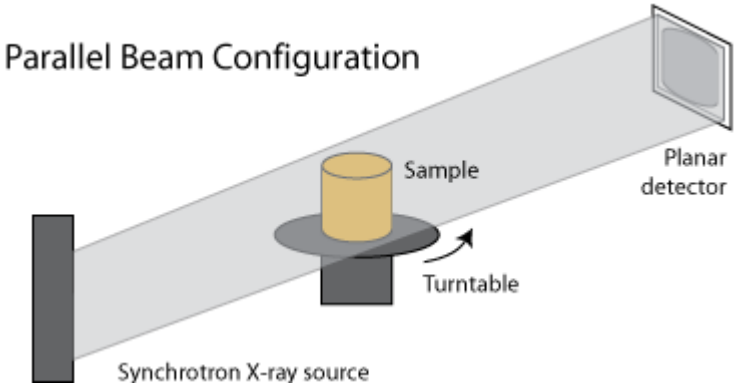
Monitoring sets of X-ray projections



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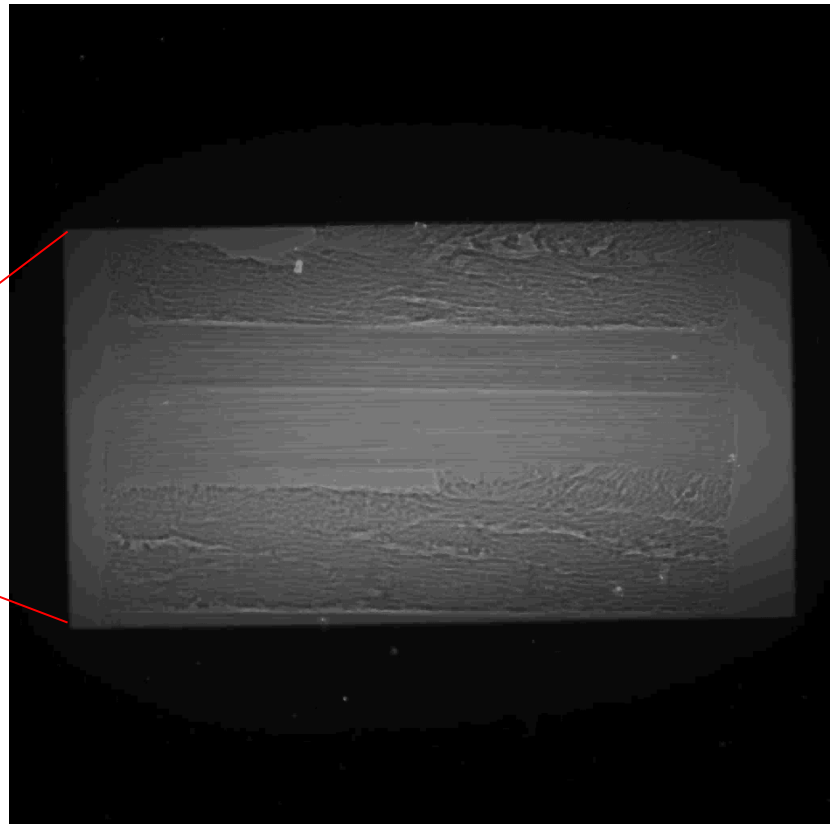

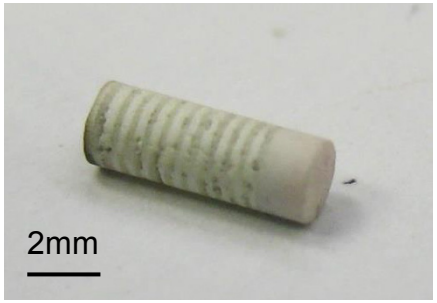

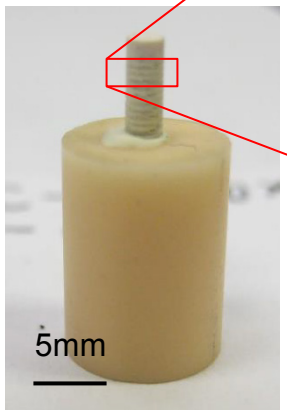

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X-ray projection

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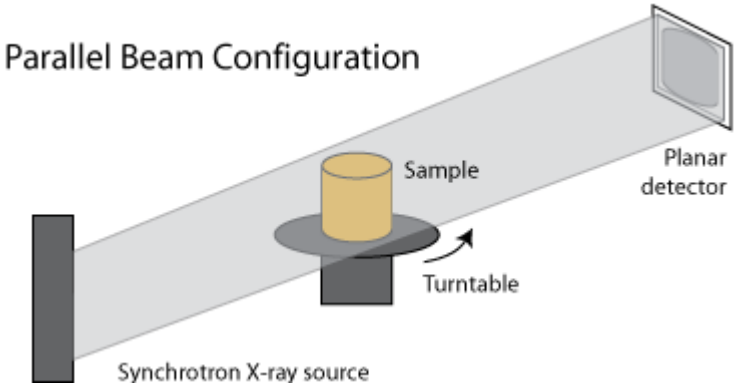
Monitoring sets of X-ray projections



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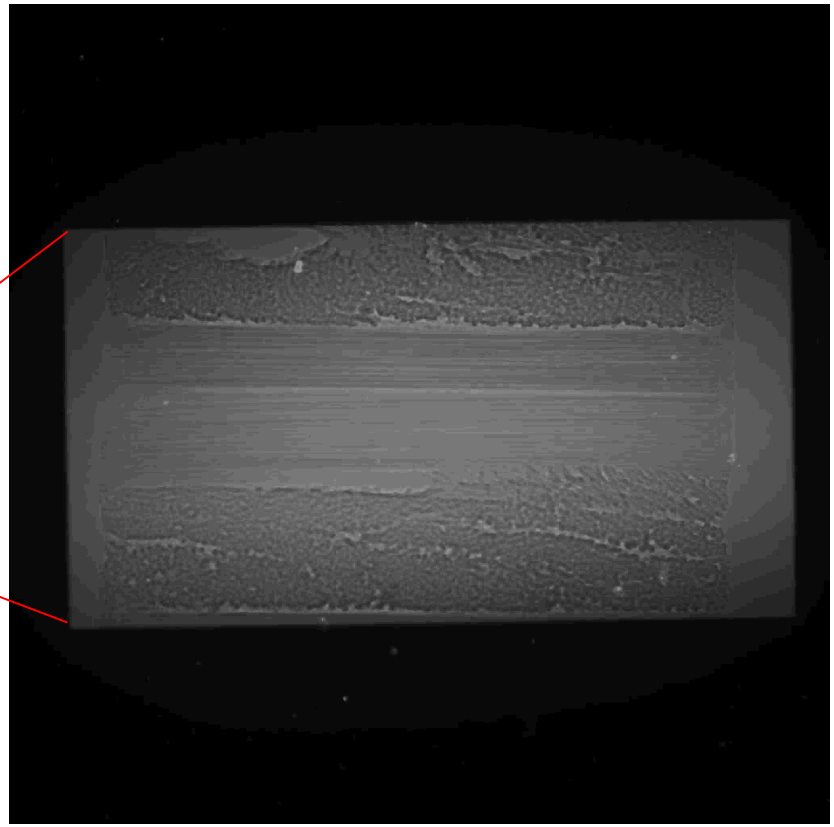

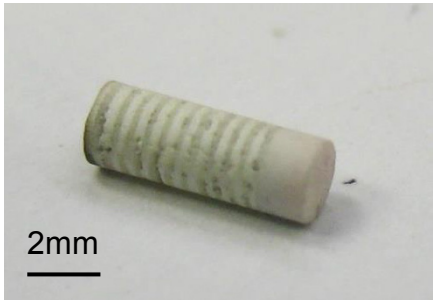

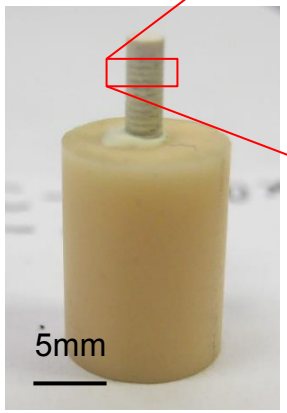

Sample

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X-ray projection

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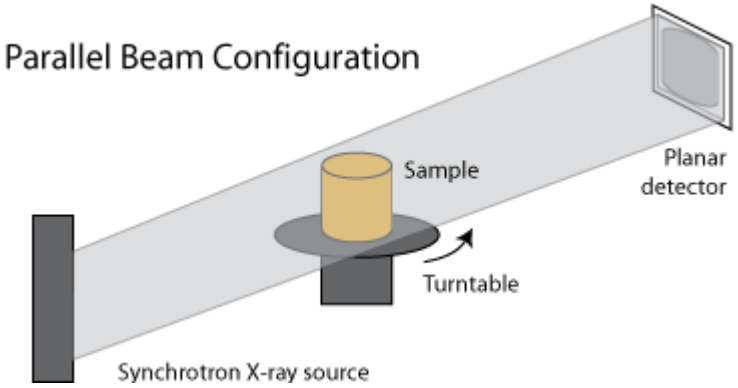
Monitoring sets of X-ray projections



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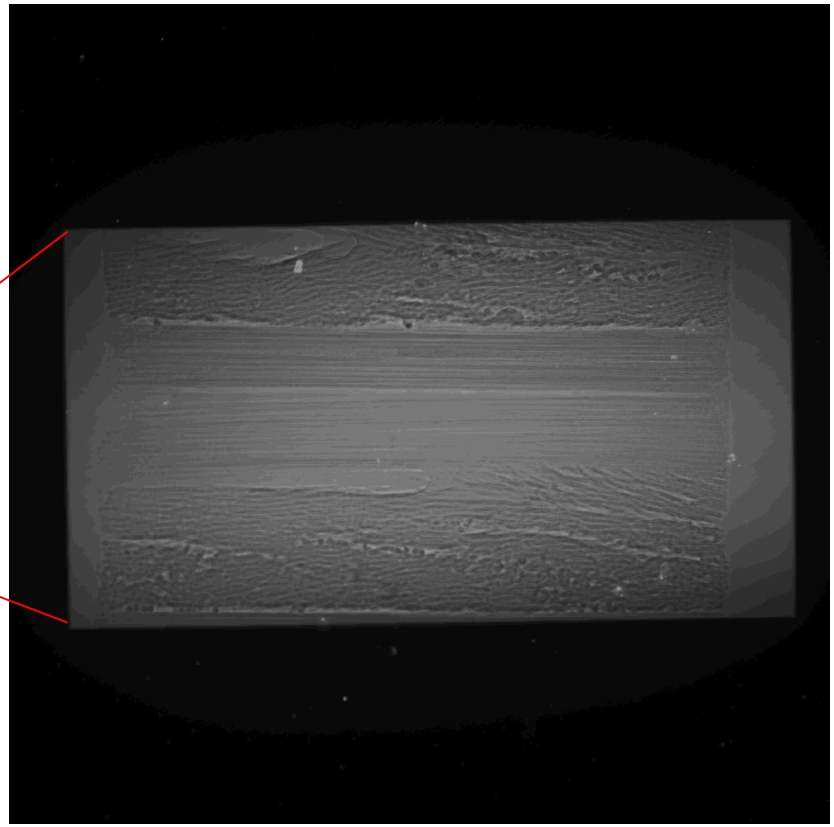
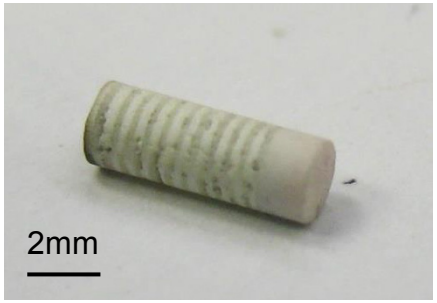
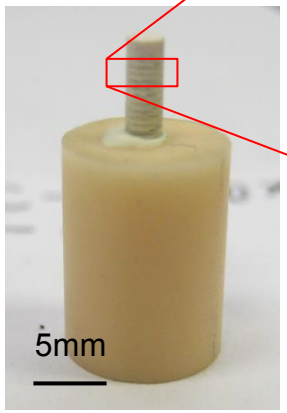






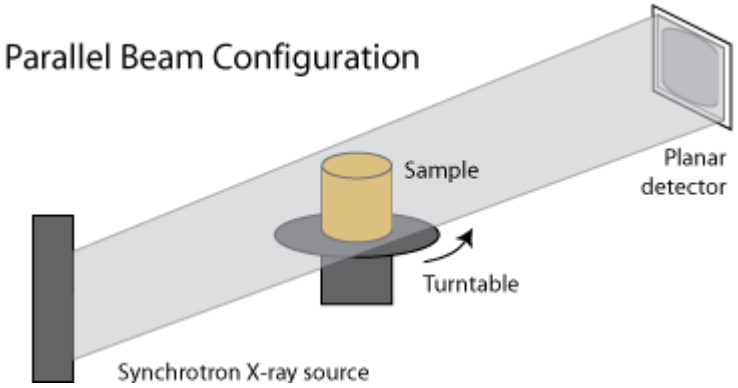
Sample

Mounted Sample

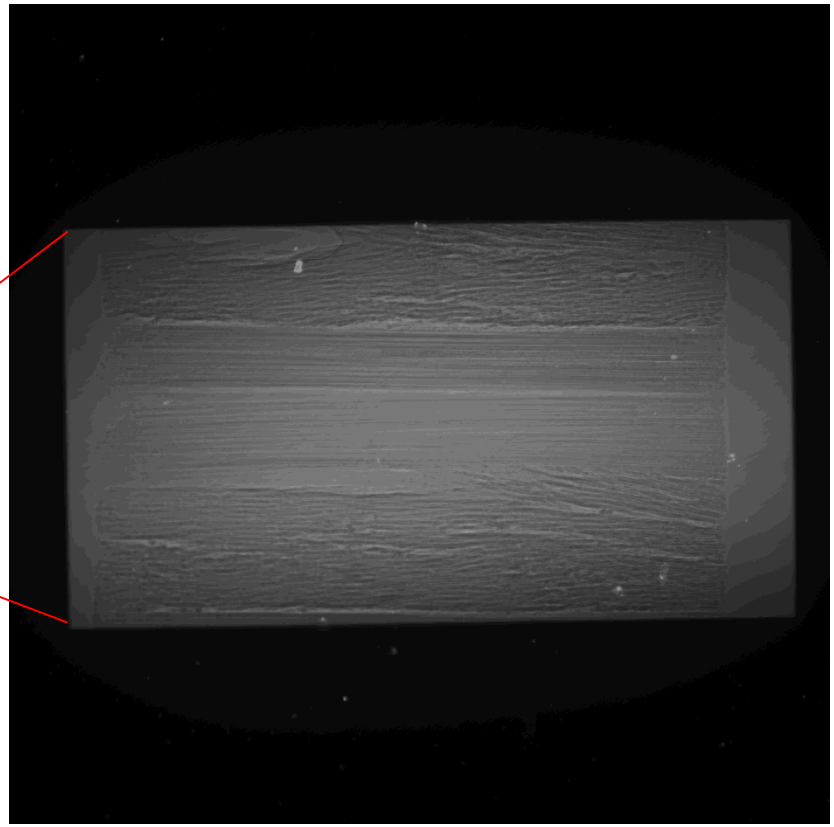
X-ray projection

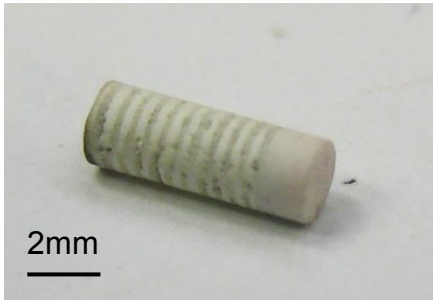
DLR

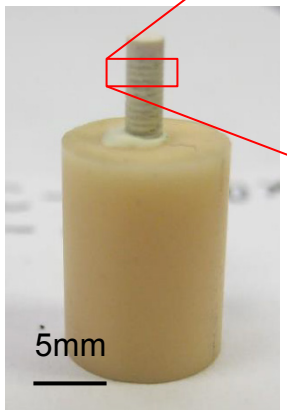
Monitoring sets of X-ray projections



Laminate with +/- 45° fiber bundles







Sample

Mounted Sample

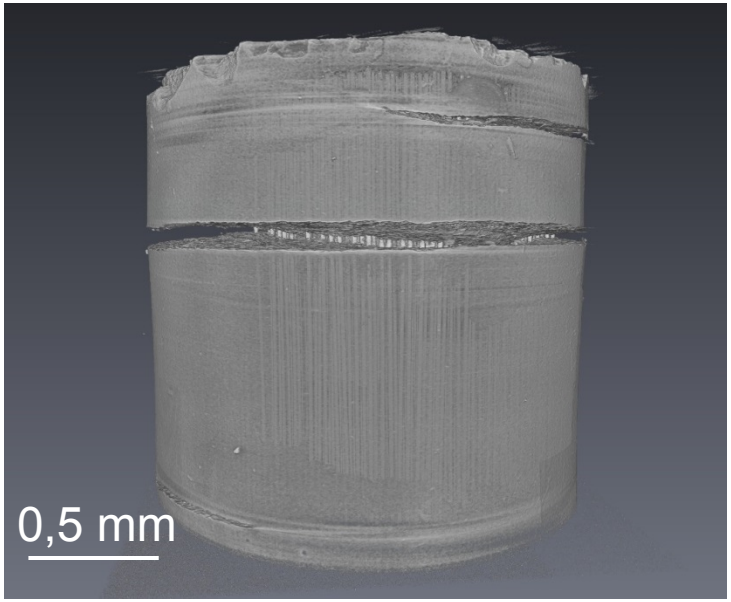
X-ray projection

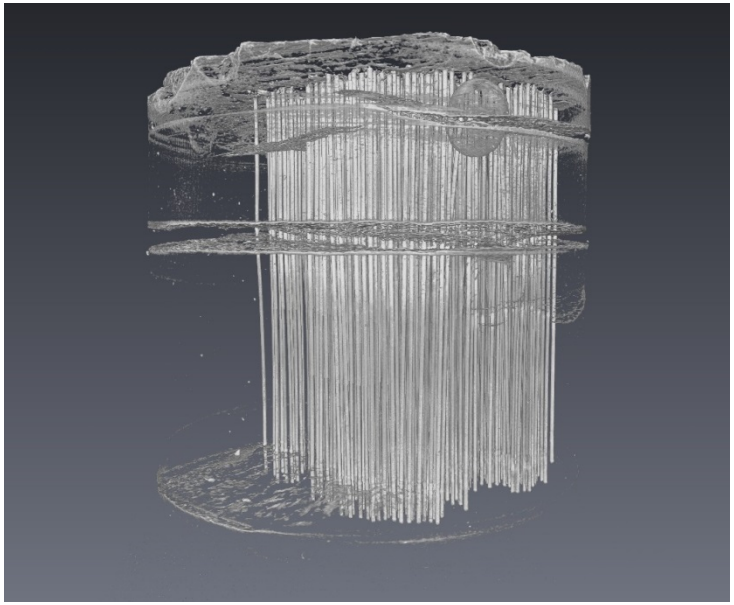
DLR

Sample Overview

Sample Nr.	Fiber architecture	Chemical composition
1	UD-single bundle	Al_2O_3-fiber, mullite matrix ($3Al_2O_3 2SiO_2$ or $2Al_2O_3\ SiO_2$)
2	UD- laminate as processed (1300°C, 1h)	Al_2O_3-fiber and matrix; as processed
3	UD- laminate aged (1450°C, 1h)	Al_2O_3-fiber and matrix; aged
4	+/- 45° laminate, aged	Al_2O_3-fiber and matrix

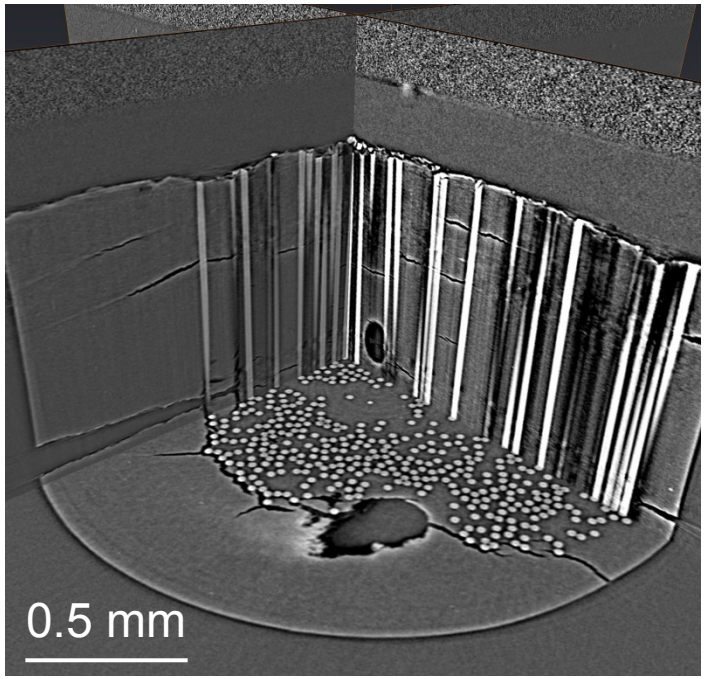
Unidirectional fiber bundle embedded in porous matrix Al_2O_3 – fibers and Mullite matrix



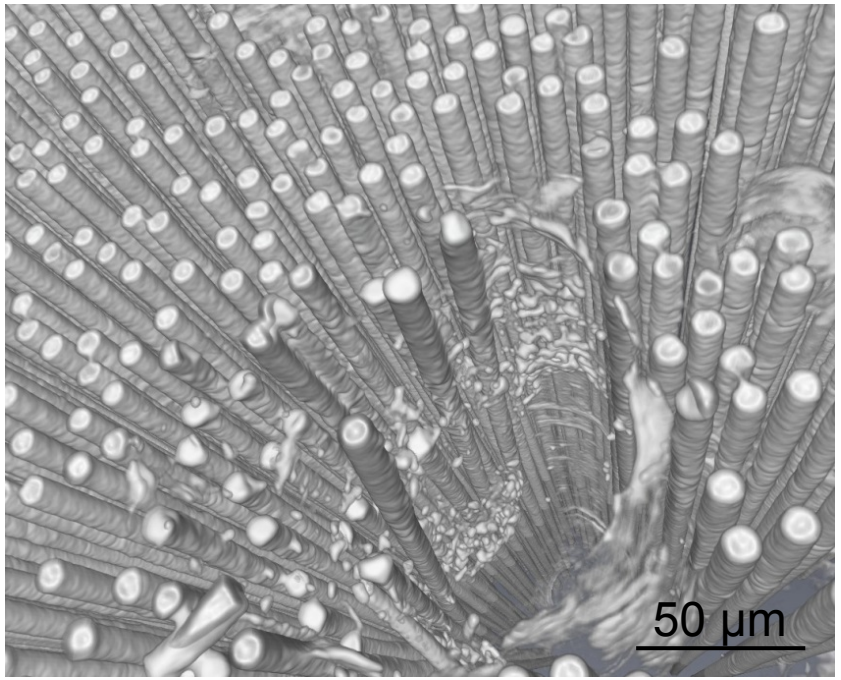


As processed: sintered 1h at 1300°C
Shrinkage cracks visible and good separation between fibers and matrix

Unidirectional fiber bundle embedded in porous matrix Al_2O_3 – fibers and Mullite matrix

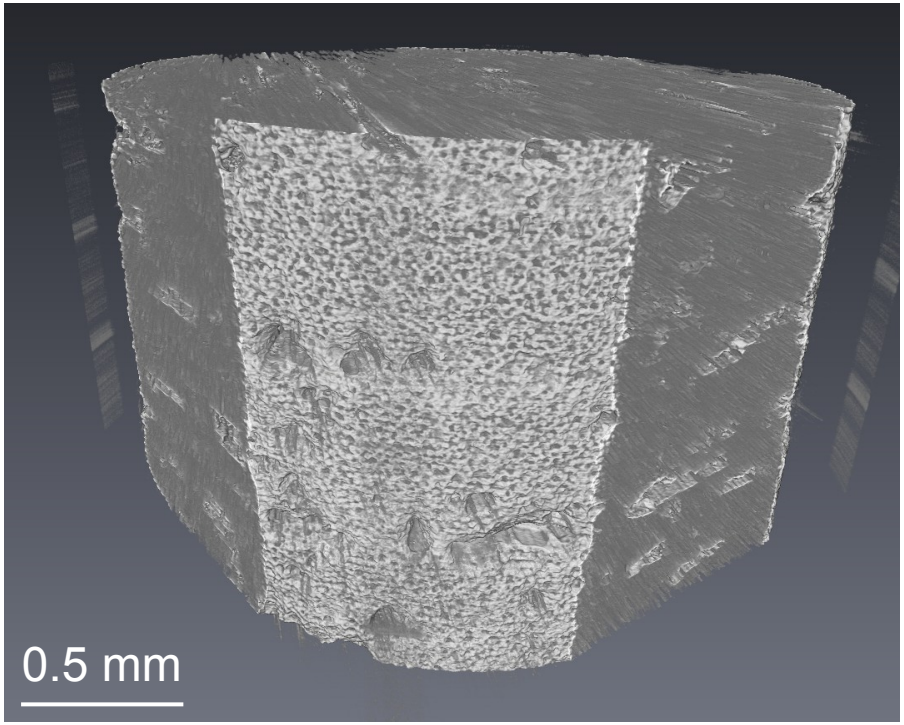


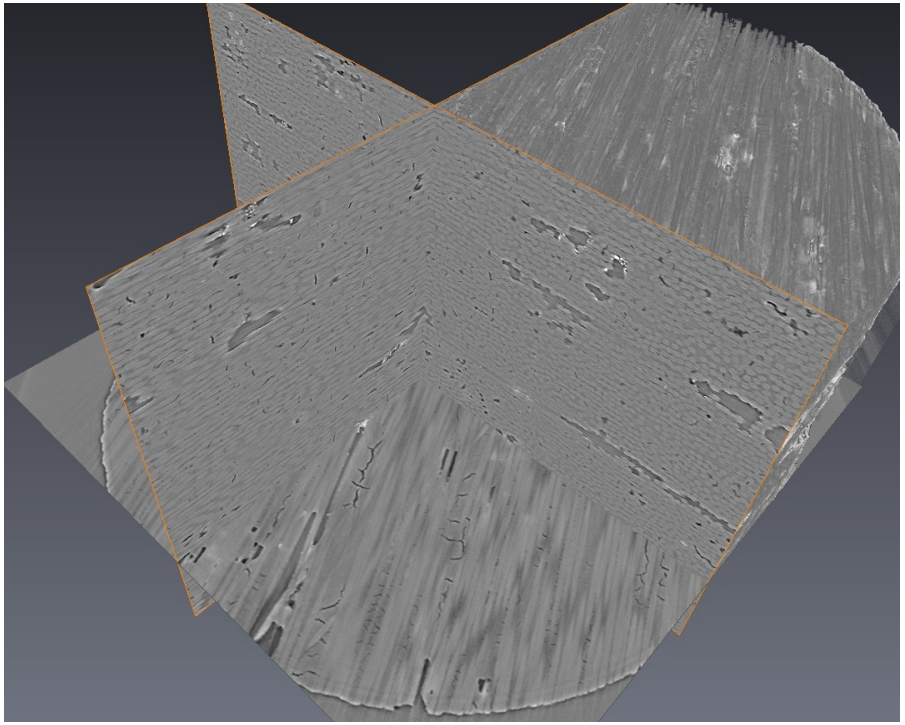
Shrinkage cracks due to processing



Fibers, matrix not shown

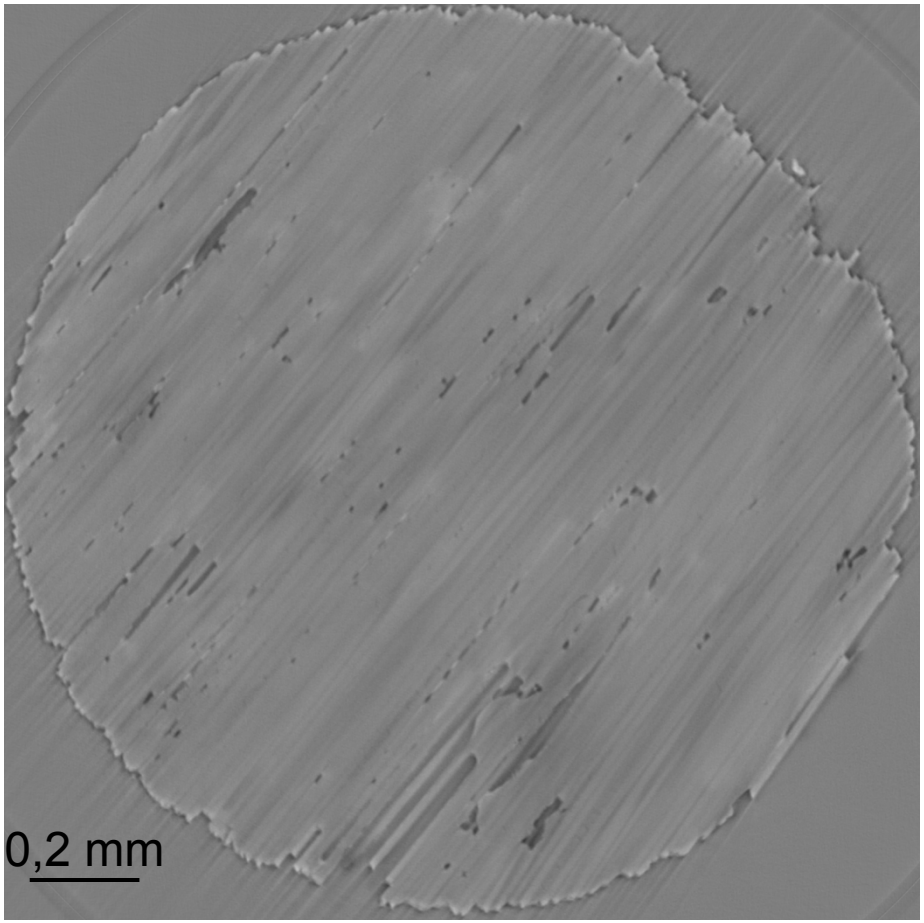
Unidirectional WHIPOX™- Laminate
Al_2O_3 – fibers and matrix (aged 1h at 1450°C)



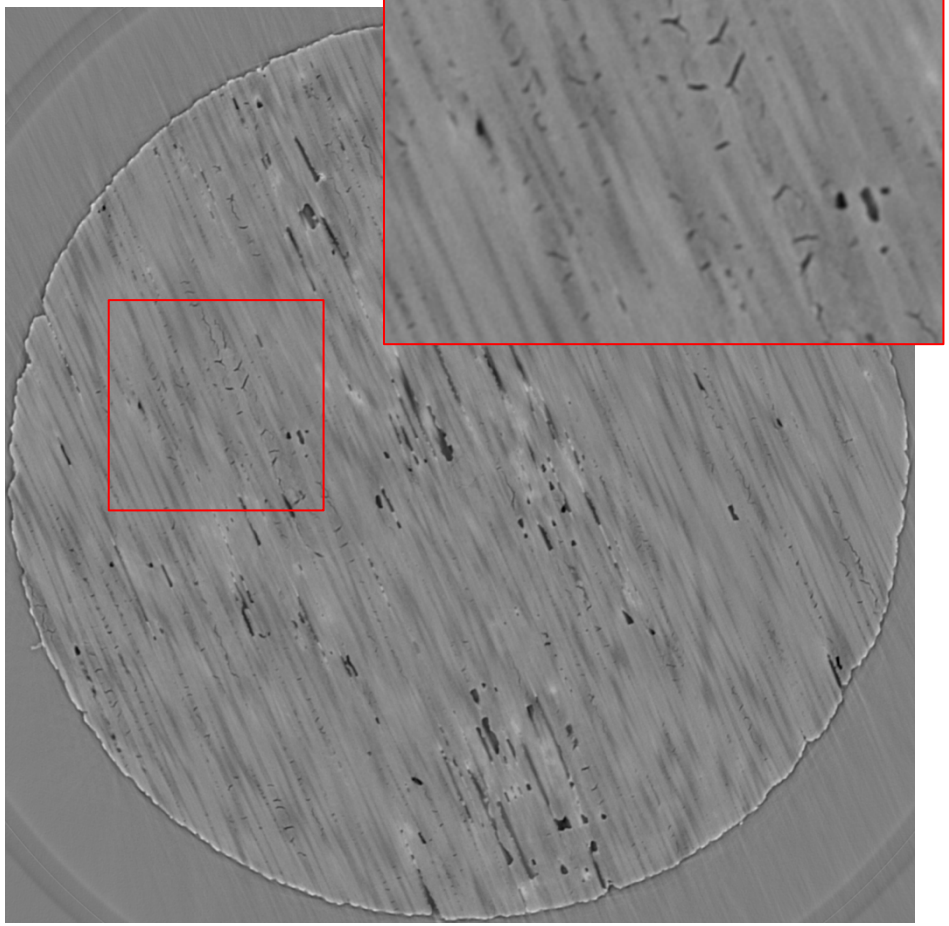


Pores in sub - millimeter range, matrix rich regions between fiber bundles, shrinkage cracks in matrix, difficult separation between fibers and matrix

Difference between as processed and aged

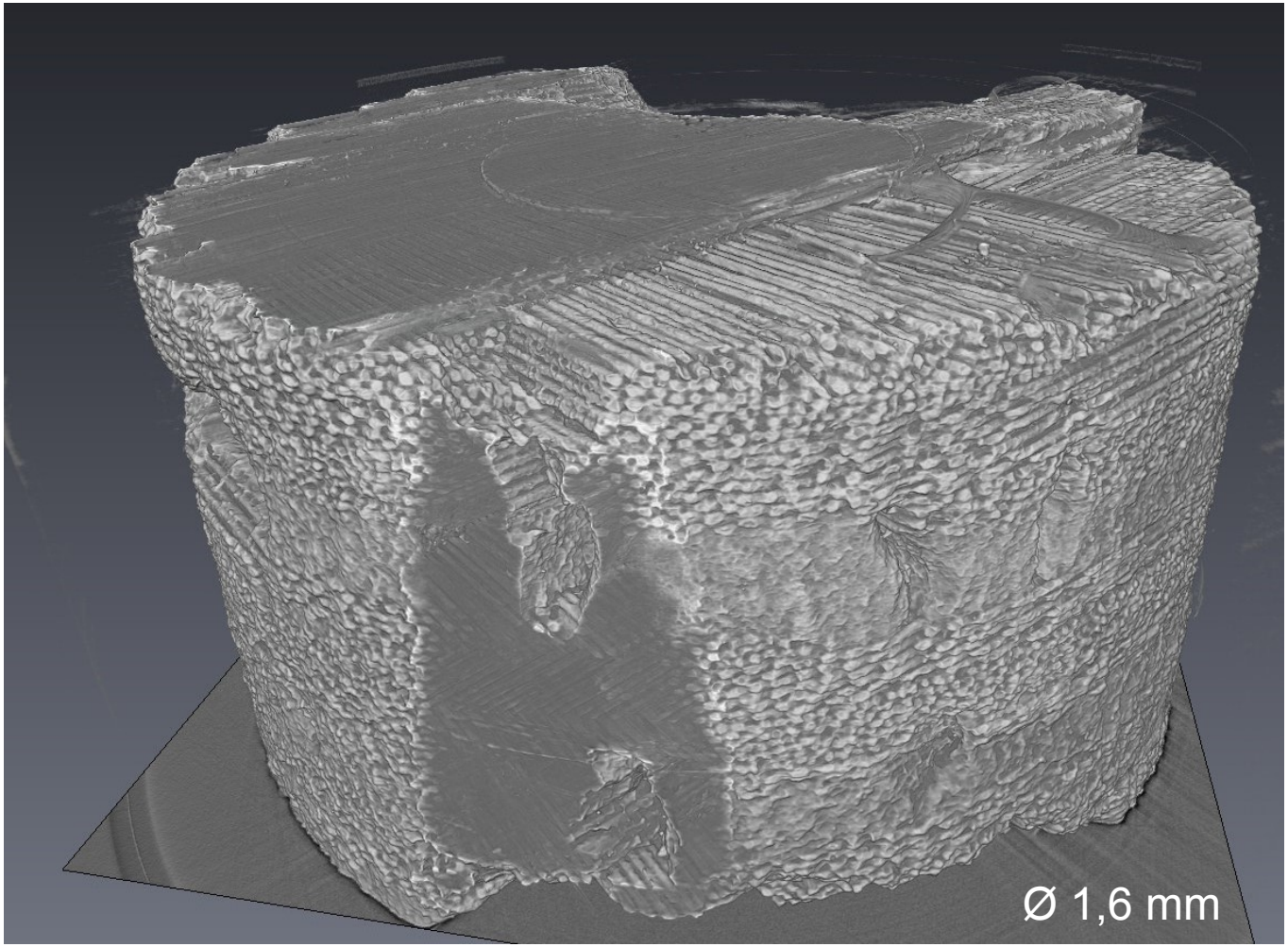


As processed – few matrix cracks



Aged – abundant matrix cracks

Laminate with +/- 45° layers (Al_2O_3-fibers and matrix)



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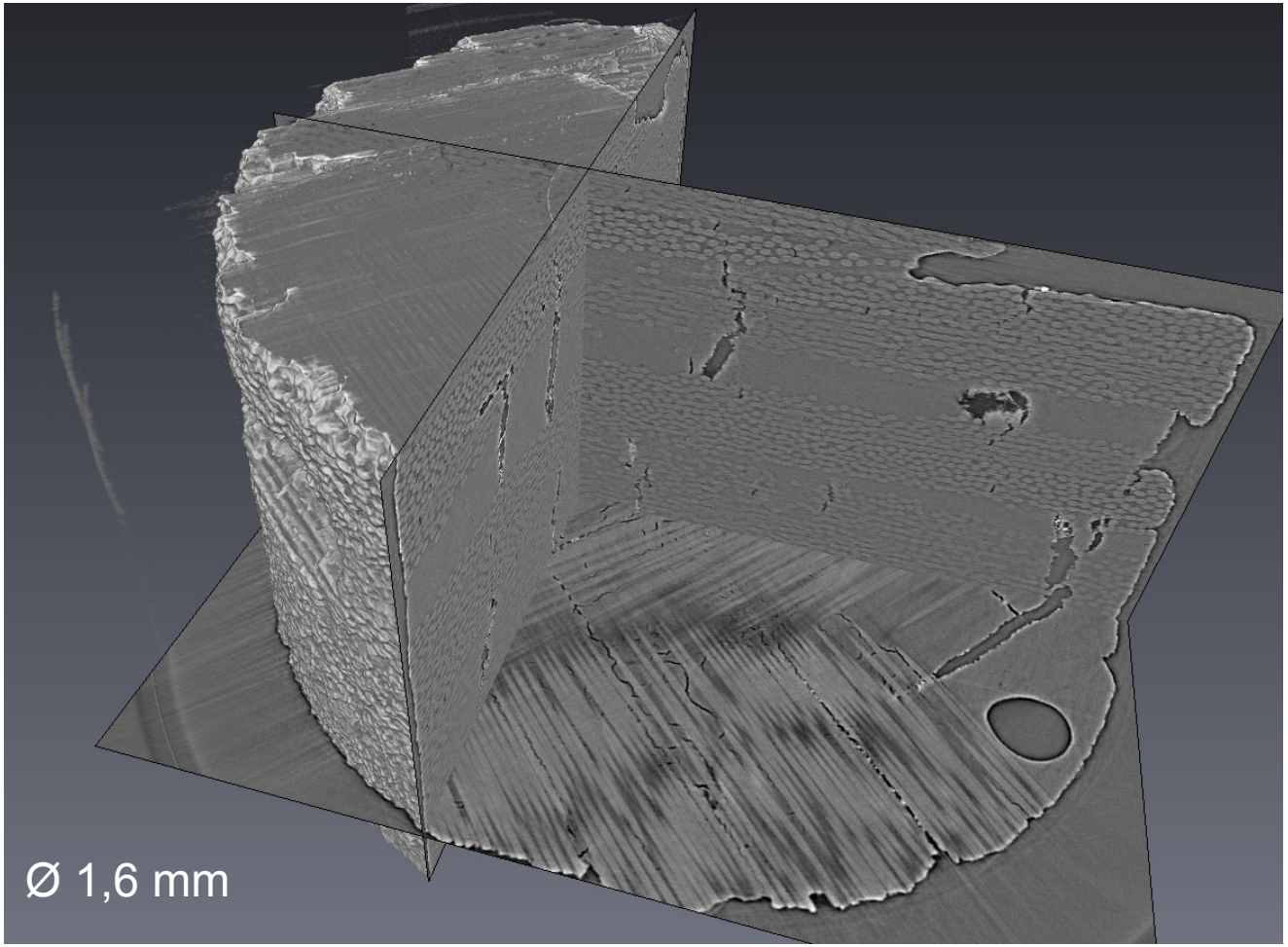
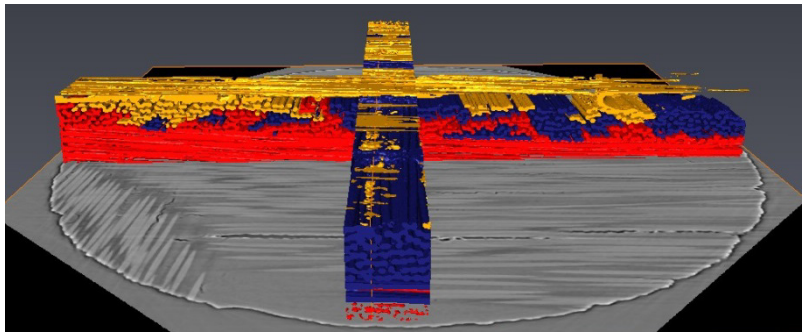
Laminate with +/- 45° layers (Al_2O_3-fibers and matrix)

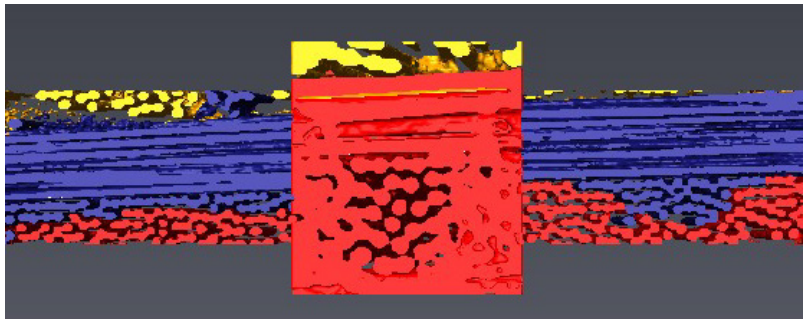


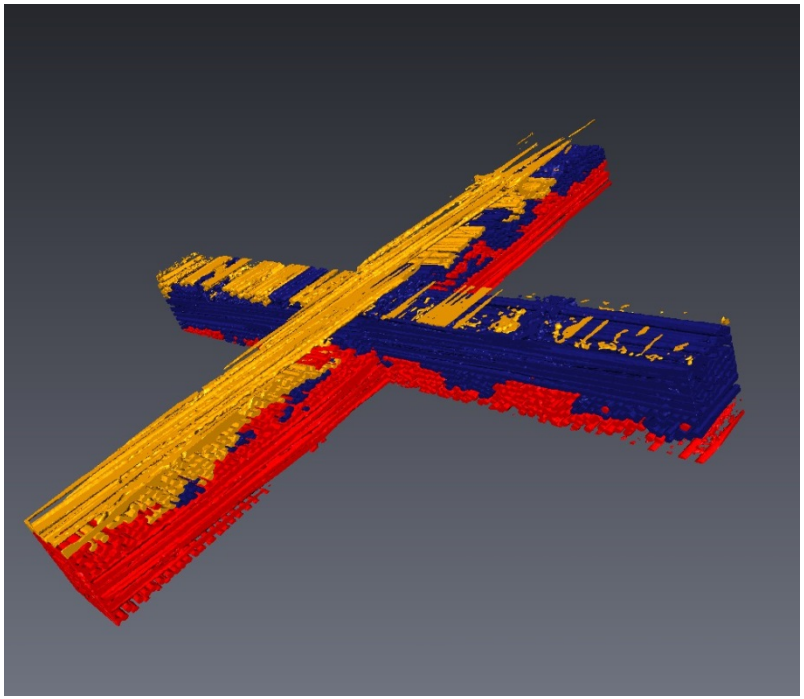
Aged at
1450°C for 1 h

Fiber bundles, sub – millimeter pores, shrinkage cracks, fiber bundle cross lines

Laminate with +/- 45° layers (Al_2O_3-fibers and matrix)

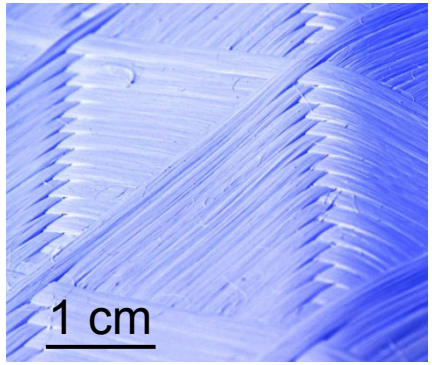






Aim: parametric description of cross line geometry
(fiber flexure, spatial fiber – matrix distribution)

Challenge: Difficult fiber separation in all alumina CMC



DLR

Laboratory X-ray tomography for high resolution

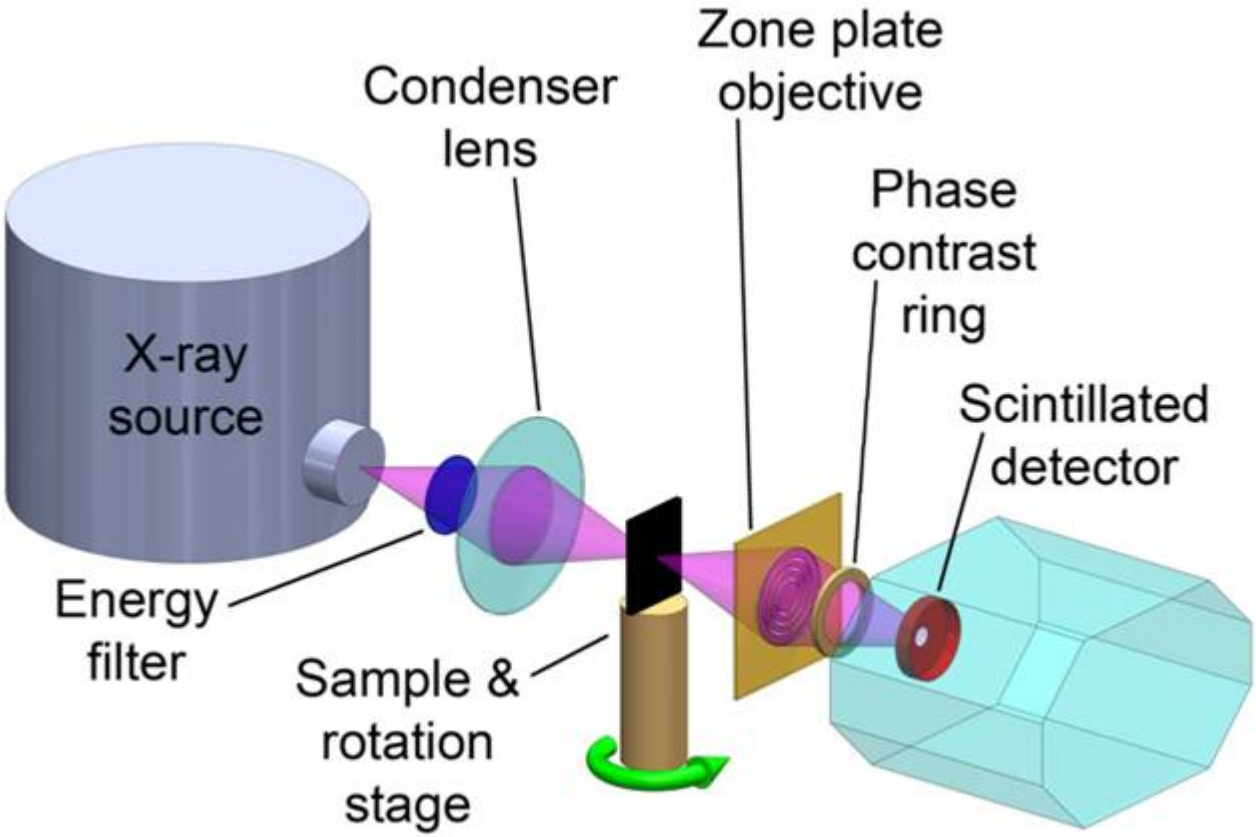
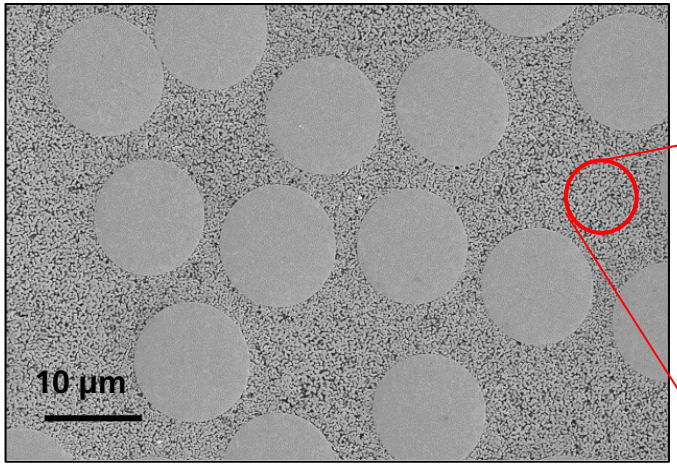


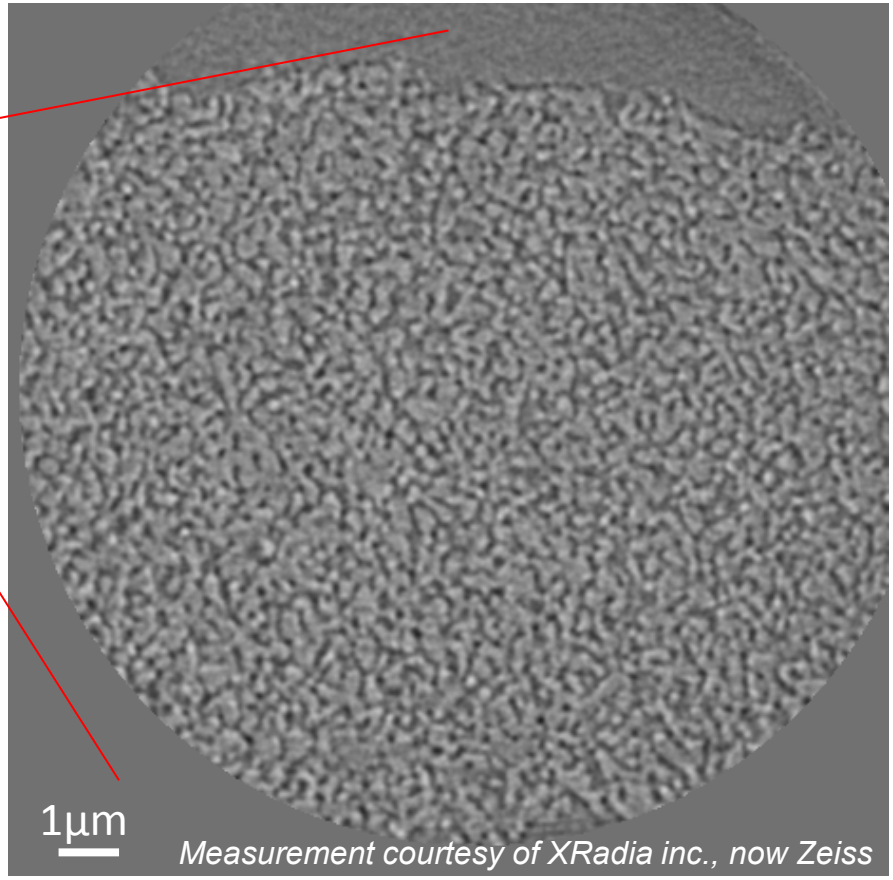
Illustration by XRadia inc., now Zeiss

X-ray tomography with laboratory devices: Porous Matrix of CMC

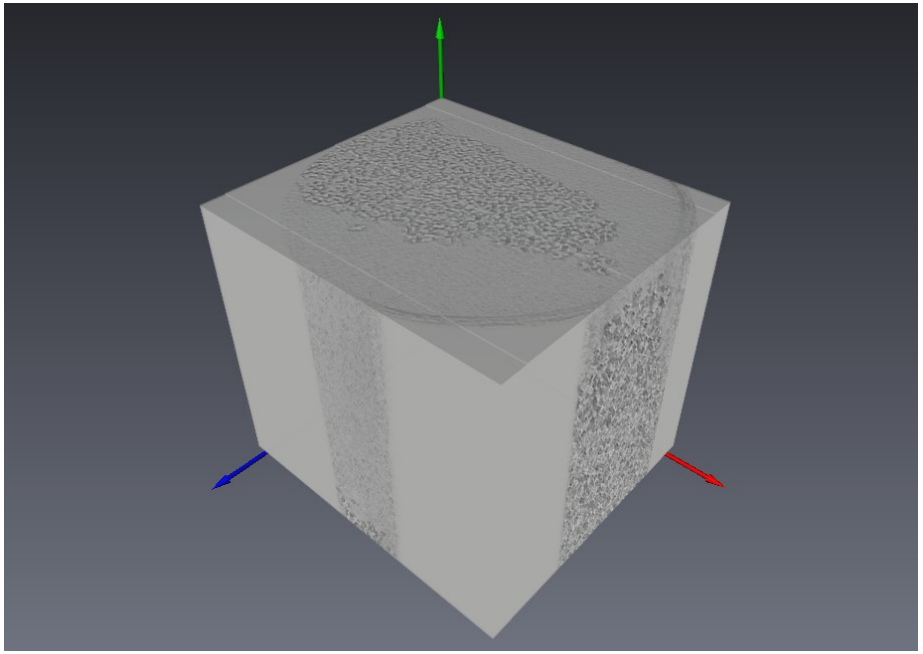


Cross section image by scanning electron microscope

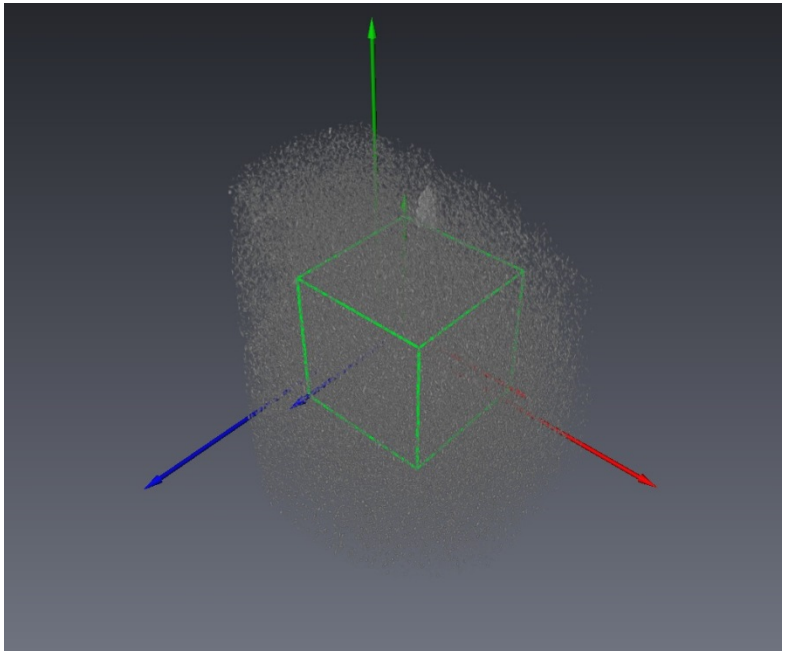
Slice of 3d-reconstruction from X-ray projections



From reconstructed X-ray data to Finite Element Model

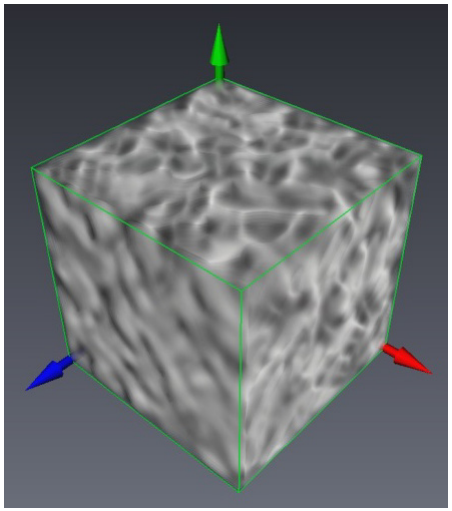


Reconstructed sample volume

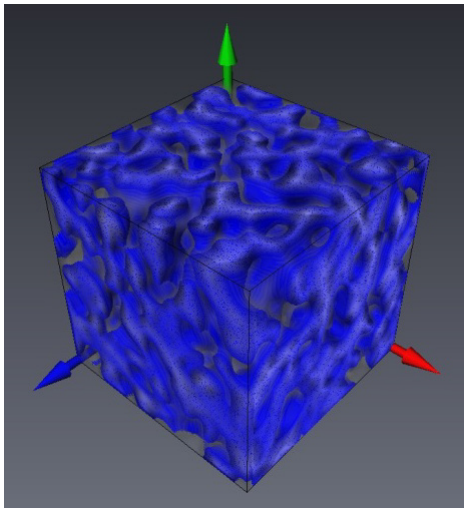


Cropped volume for analysis

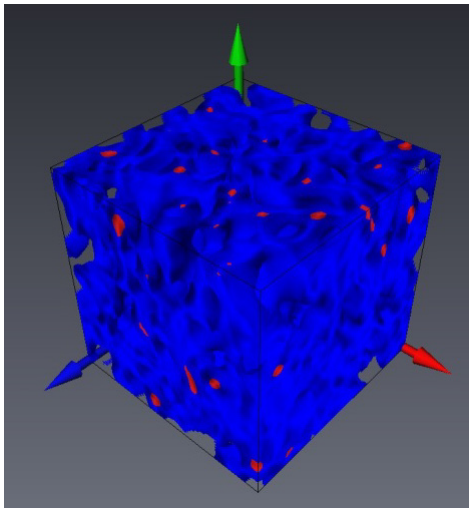
From reconstructed X-ray data to Finite Element Model



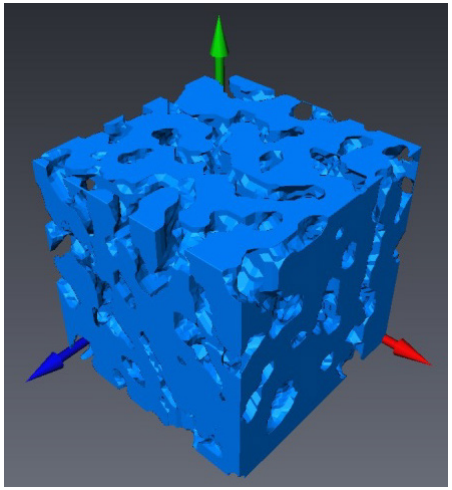
Cropped volume



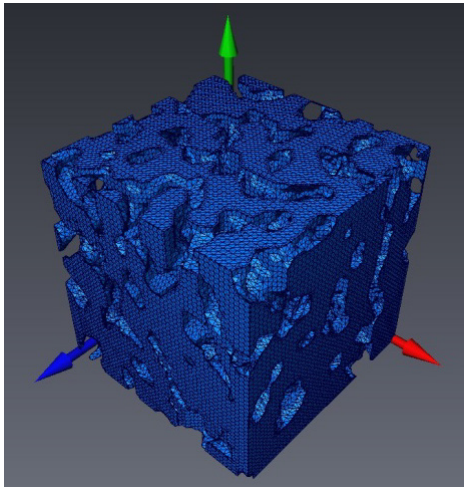
Segmented pores



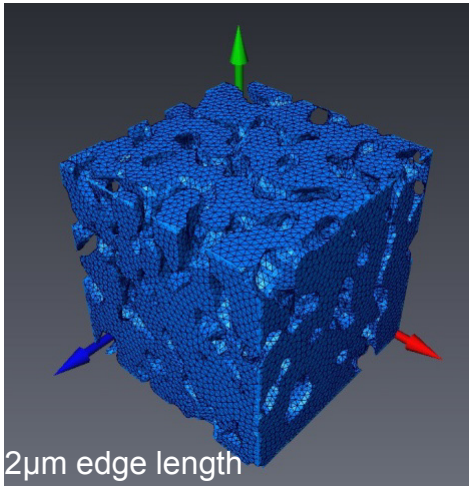
Segmented ZrO_2 particles



Surface model

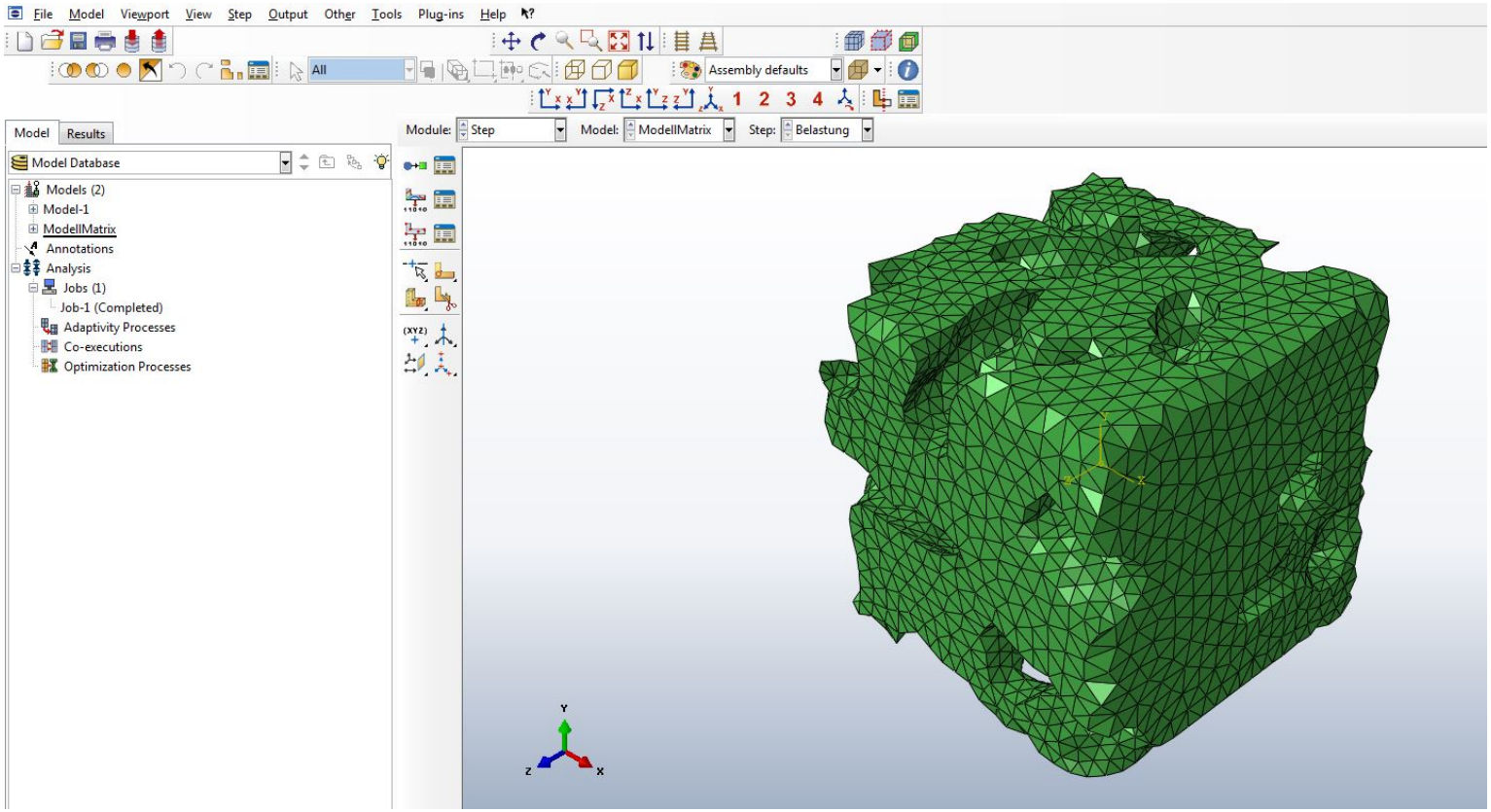


Meshed surface

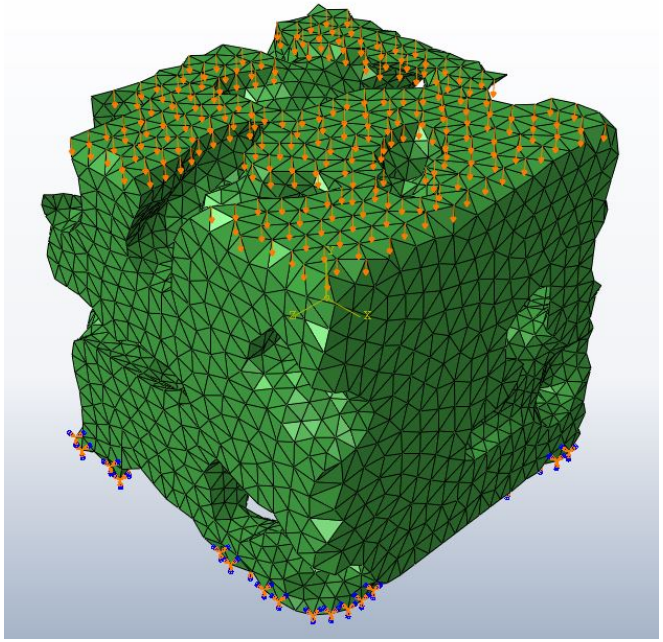


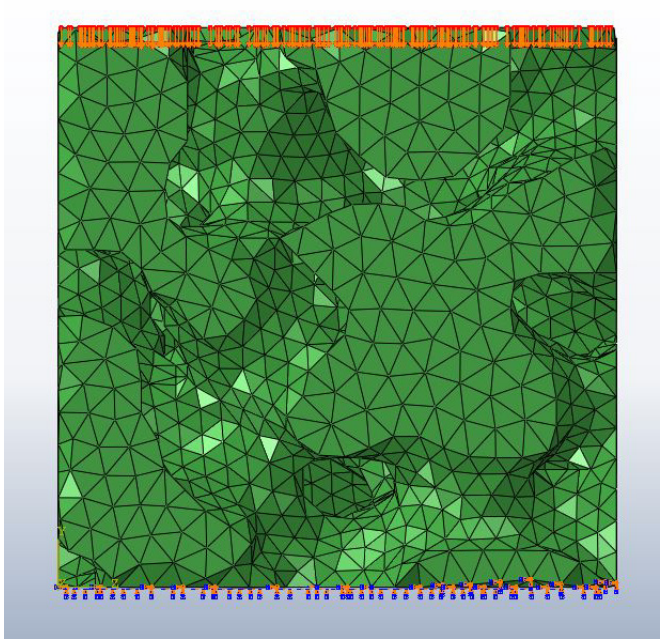
Meshed volume

Import of 3d-image into Numerical Calculation Tool



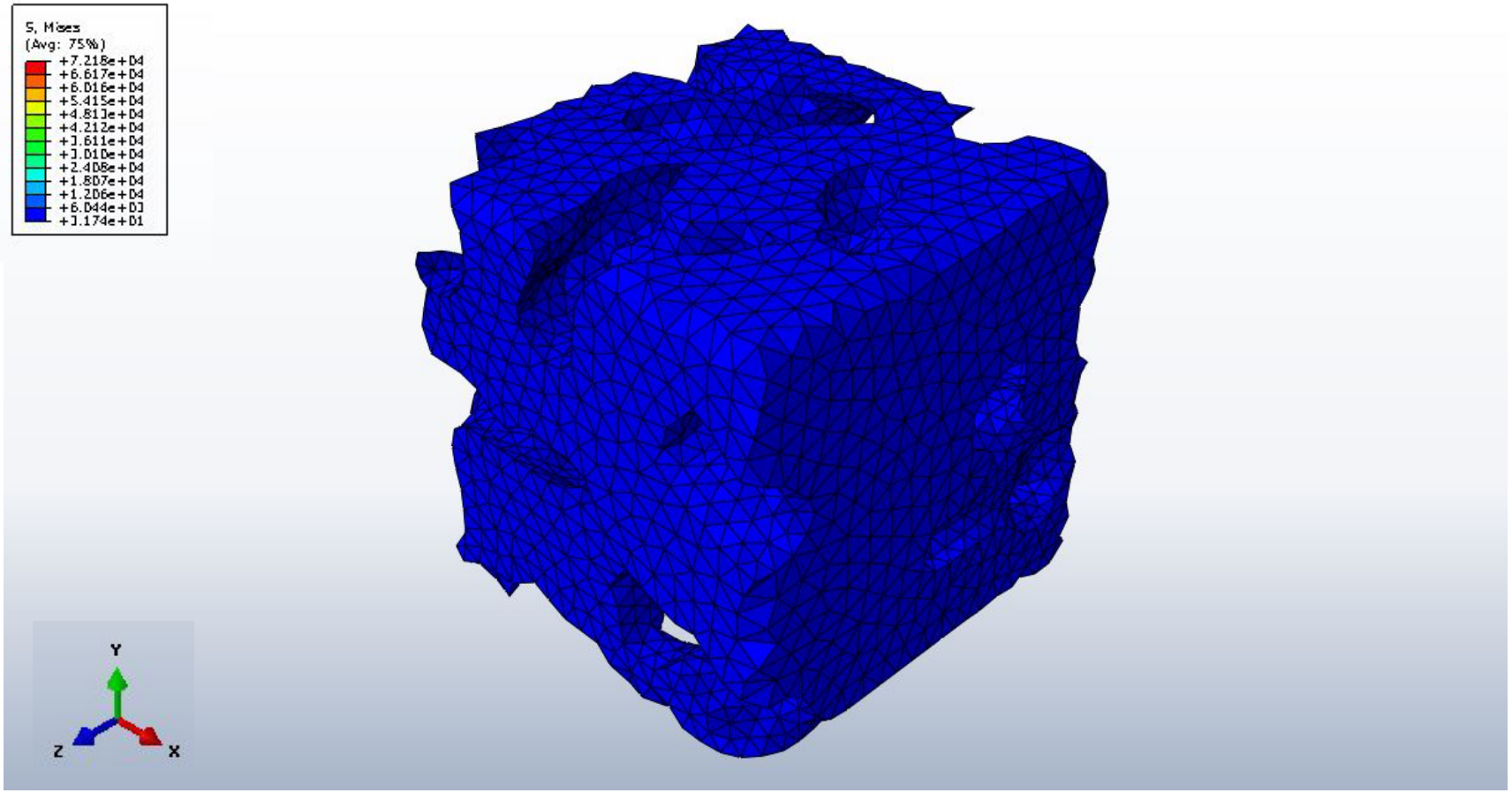
Exemplary virtual mechanical experiment



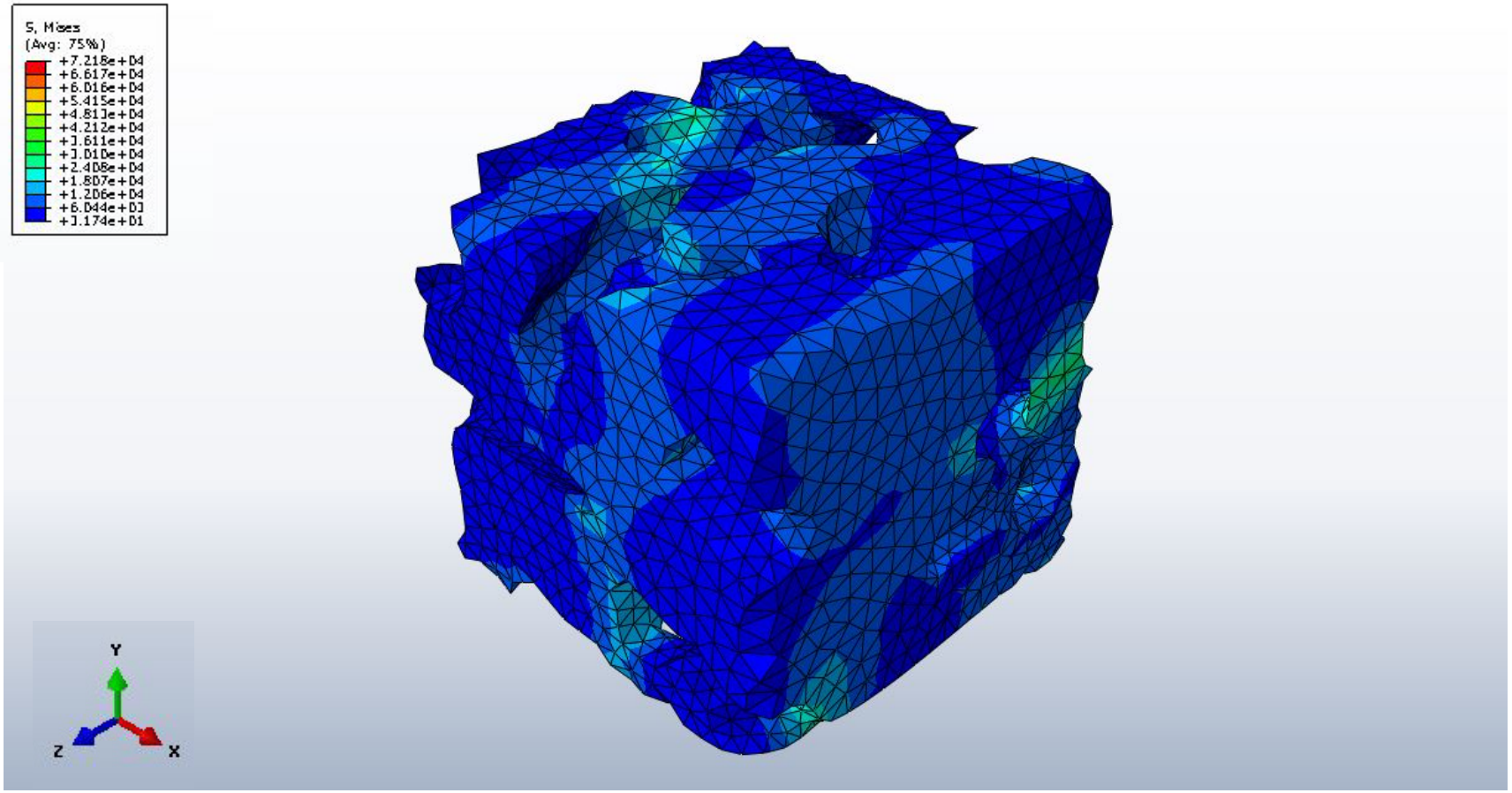


Generating compressive stress by displacement of top plane

Exemplary virtual mechanical experiment



Exemplary virtual mechanical experiment

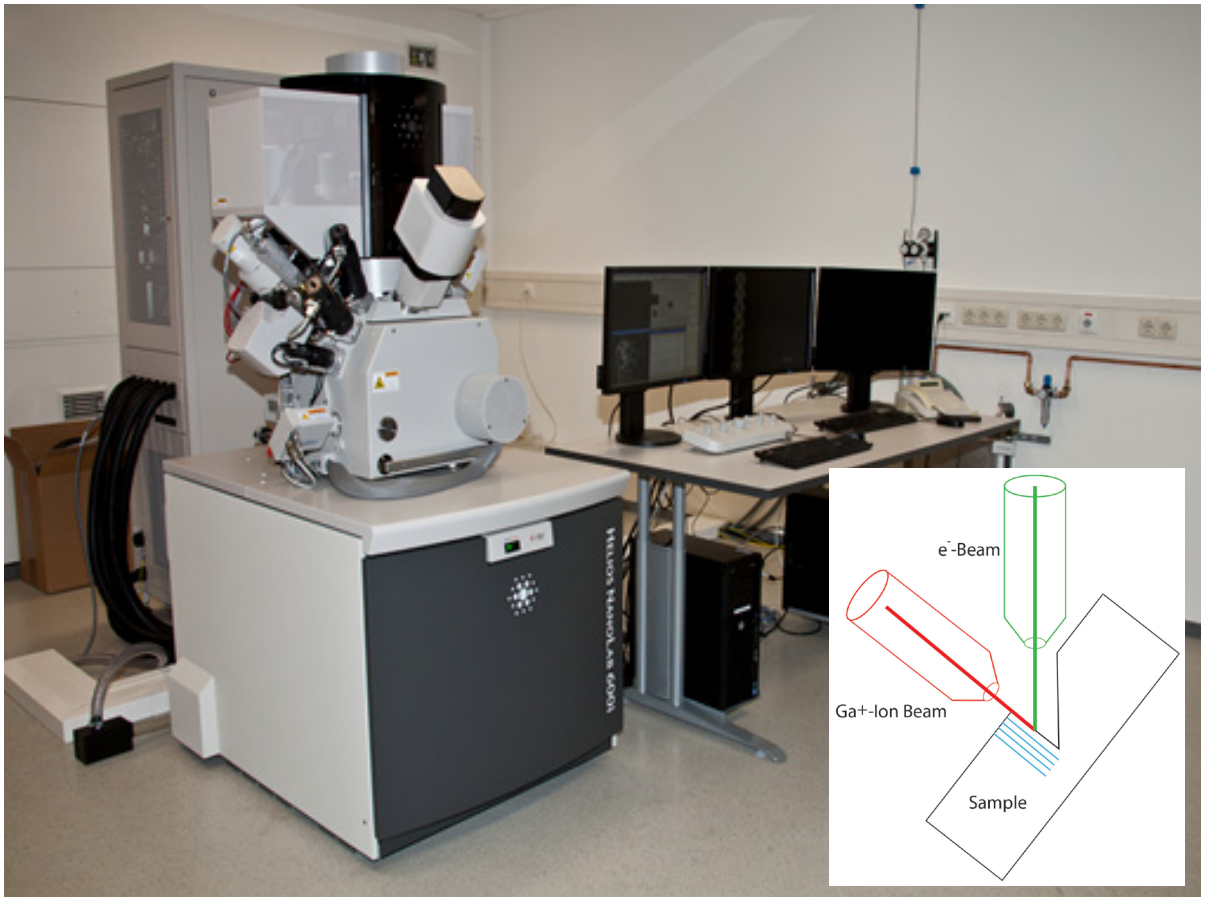


Tomography based on FIB-Slices

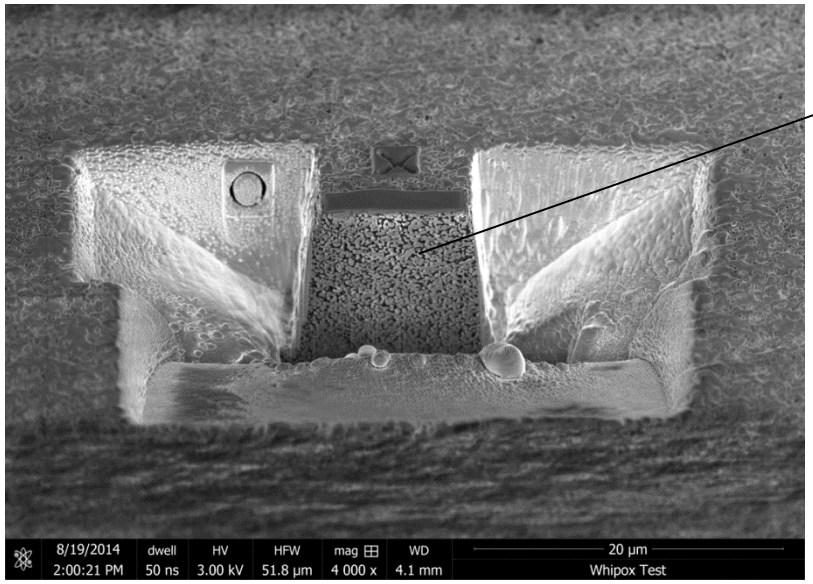
FIB: Focused ion beam

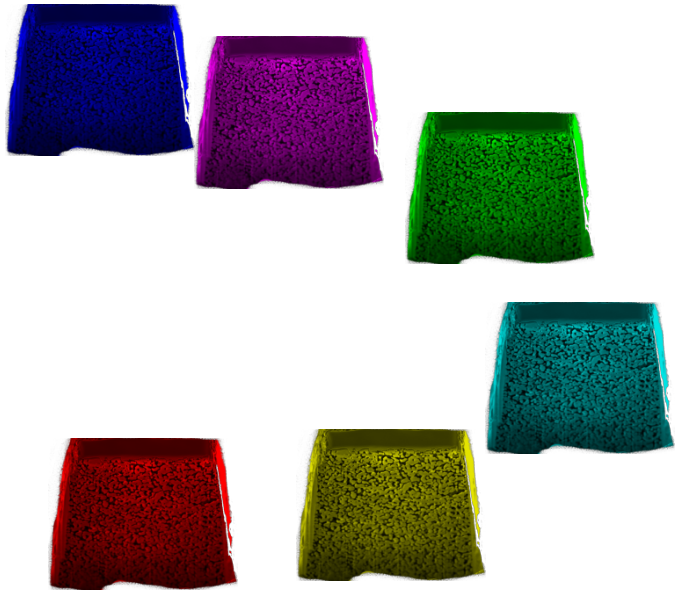
SEM: Scanning electron microscope

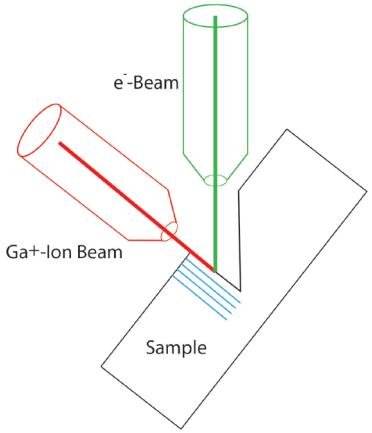
Generating 2d-slices by ion beam cutting and subsequent recording SEM-images



Generating a 3d – image of porous ceramic



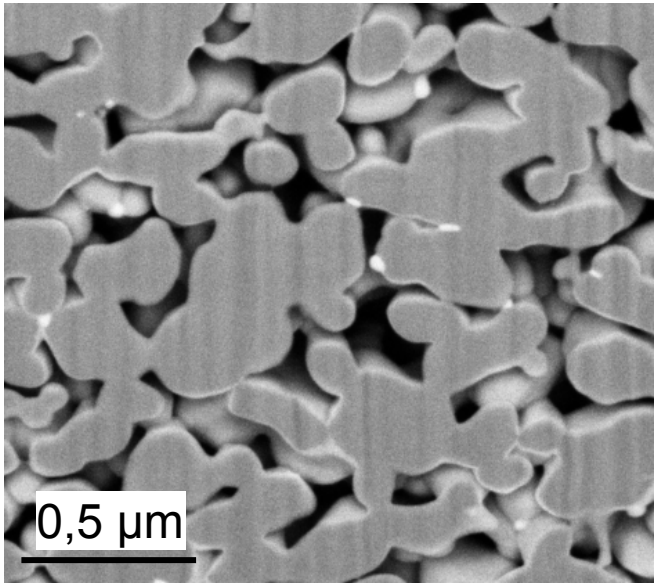


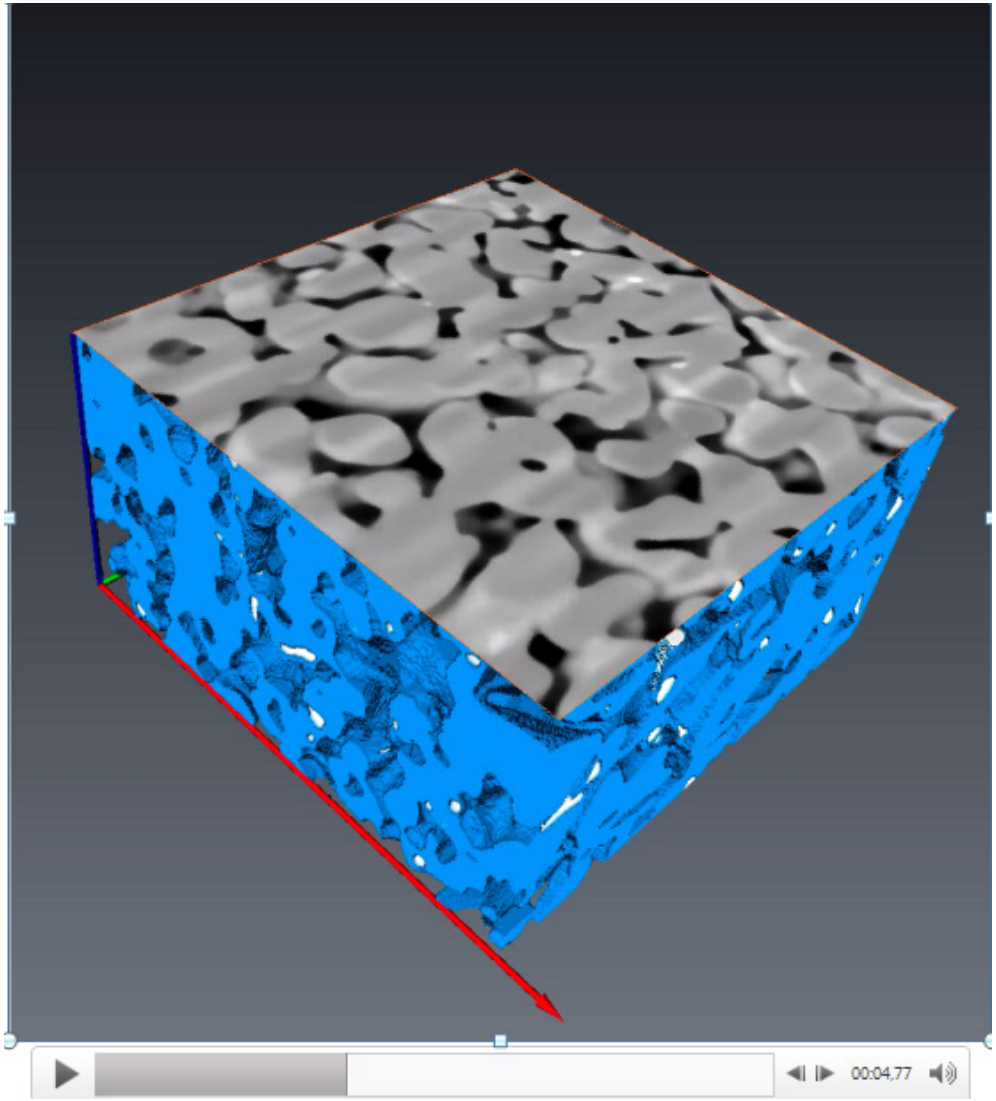


- alignment
- scaling
- cropping
- segmentation

➔ 3D-Volume

Generating a 3d – image of porous ceramic II

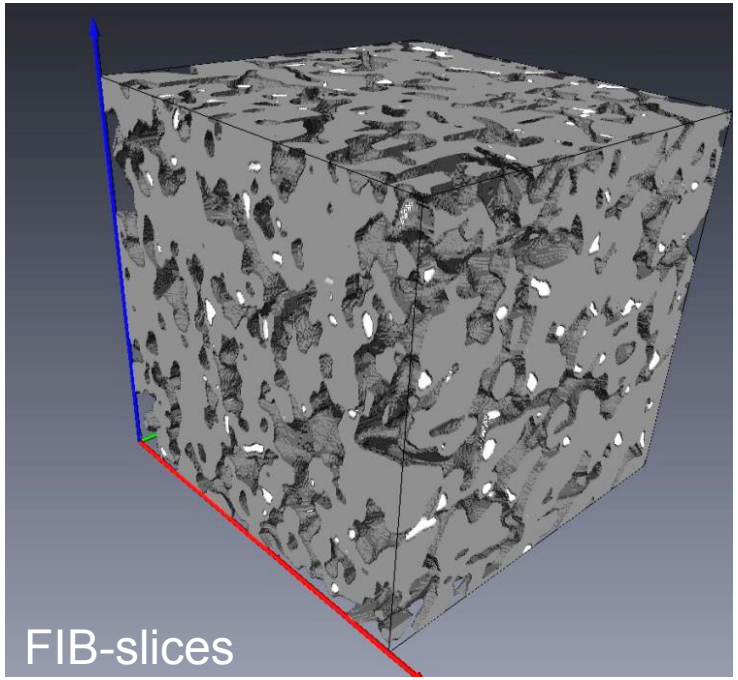


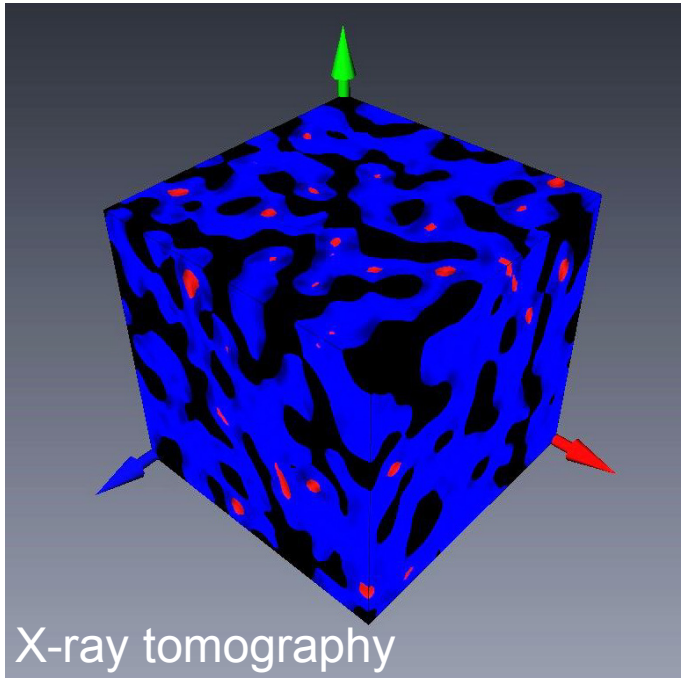


Challenge: segmentation of pores and matrix due to information from underlying planes

DLR

Porosity from 3d-images and by Archimedes method





Method for porosity measurement	Porosity %	ZrO_2-Vol. %
X-ray tomography	35 - 36	2-3
FIB-slice tomography	35,4	2,8
Archimedes	36,1	-

Summary and Outlook

- X-ray tomography can provide excellent 3d-images of microstructures down to sub-micron range
  - Non destructive technique
  - Using high energy synchrotron X-ray radiation provides unique options:
    - Short acquisition time
    - No (severe) restriction of sample size for high resolution images
      - Acquisition at high temperature possible
      - Acquisition under mechanical load (in-situ testing)
  - Using laboratory devices with x-ray point source (and additional x-ray optics) allows high resolution but sample size is restricted (ca. 50µm)
- Slice and view technique using focus ion beam and scanning electron microscope allows higher resolution
  - But no in situ acquisition at high temperatures or under load
  - Possibility of generating artefacts when slicing
  - Challenge: porous samples, showing in each slice information from underlying material

Summary and Outlook II

- Data from X-ray tomography and slice and view techniques can be used for generating numerical microstructure models
- Virtual experiments can be done with Finite Element software
- Image analyses can be used to identify geometry parameters of the microstructure, which allow for generating virtual microstructures
    - Parameter studies for elucidating the microstructure - property relationship

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Questions?

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